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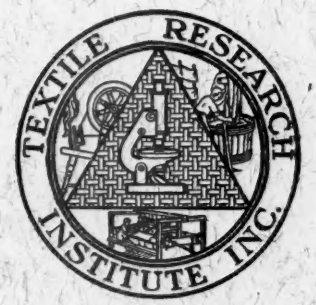

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TEXTILE RESEARCH JOURNAL

VOL. XVIII, No. 3 March, 1948

A Generalized Concept of Resilience

R. M. Hoffman

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Different Kinds of Resilience

Resilience means different things to different persons. This is apparent in a survey of the literature made by Dillon [1] and can be further illustrated by the following viewpoints: A rubber manufacturer might say, "Rubber is resilient," and a textile manufacturer could say, "This fabric is not very good; it is too rubbery." A tire expert could say: "I need a high work recovery * in tire cord to avoid generation of heat at high speeds. An experimental tire made of woolen cords overheated badly; therefore, wool is not resilient."

A physicist says: "The behavior of a quartz ball dropped on a quartz plate provides a good example of resilience; it will bounce many times, showing a small loss of energy through dissipation as heat. In other words, it has a high work recovery." But a carpet manufacturer can say: "The resilience or 'luxury' factor of a carpet is proportional to the reciprocal of the modulus. I want a low modulus. A quartz plate takes a 'zero' on my resilience scale because it won't absorb any work in compression."

Such statements, when taken together, constitute a paradox because they are all at least partially true but, at the same time, inconsistent. It is believed that such contradictions can be reconciled by a generalized concept of resilience.

An analysis of the available information indicates that a common denominator exists for resilient materials of all types. This common denominator is: a high percentage of recovery after, but not necessarily immediately after, a deformation. Resiliency † can be thought of, therefore, as the capability of a substance to return to its original state at some later time after the removal of a deforming stress. Such a statement is deceivingly simple. Its value lies in the fact that it provides a basically sound starting point for distinguishing between the *extent* and the *kind* of resiliency.

It is impossible to study the resiliency problem without sooner or later coming to feel that not only do the various materials differ in extent of resiliency, but also in kind. Even though a resiliency coefficient of 99 percent might be assigned to both quartz and rubber, one is left with an uncomfortable

* Ratio of useful work returned by system to work put into system.

† The word "resiliency" is used here to mean an inherent property of a substance. "Resilience" refers to a more extensive property which takes into account the size and shape of the object as well. This distinction can be brought out more completely after first developing the general concept, and it is discussed in a later section of this paper under the heading "Resilience and Resiliency."

feeling that the two materials are really more different than such a rating would indicate. Wool is different, so wool-like resiliency is referred to as something special. But it is obviously undesirable to invent a new brand of resiliency every time a different material is encountered. The question is: How many kinds of resiliency exist? It is believed that there is a logical approach to this question which will yield a small, finite number of major types. The concept outlined below indicates that there are four main types.

If there is more than one kind of resiliency, we will no longer be handicapped by believing that it can be depicted on a "thermometer scale," with all the good materials at the top and all the poor or nonresilient materials at the bottom. It would be fairly easy to get agreement that cotton belongs near the bottom of such a scale and rayon perhaps halfway up. But it is difficult to get agreement as to whether wool, quartz, or rubber should occupy the top of the scale.

A prospective purchaser of an automobile might be interested in data which reveal something about the type of car he is being shown; for example, the horsepower rating and gas mileage. Because there are two parameters, this information could not be handled on one vertical scale, but it could be used to construct a two-dimensional graph or map. A third question of importance to a car buyer would be the cost, or the answer to the question "How much?" All of this information for a number of cars of different makes could be shown on a three-dimensional plot such as that in Figure 1.

It is believed that an analogous situation exists for resiliency. We must be able to talk about what kind and how much in comparing the resiliency of various materials.

It is generally agreed that the study of resiliency is a study of stress, strain, and time. These variables are interrelated in such a manner that they must usually be used in combinations such as stress per unit strain (modulus), increase of strain per unit time (creep), disappearance of strain per unit time (recovery), together with additional defining specifications such as rate of loading, etc. The next question is what combinations of these variables should be used for establishing the relative positions of all the various resilience materials on a contour map such as Figure 1. Eventually, for a complete understanding of the problem, a number of additional questions must be answered, such as whether the stress-strain-time measurement should be carried out in tension, torsion, bending, or compression, and whether the fibers should be tested wet or dry or both ways, and so forth.

But as a start, one can consider an experiment in which yarns made of various materials are suspended from a support, with a weight attached to each one. After a certain time the weights are removed, and the recovery behavior of each yarn is recorded during the next 24 hours. If at the end of this recovery period, several of the yarns have "come back" either very little or not at all, they obviously have very low or zero resilience. Now let us imagine that four of these yarns turned out to be completely resilient and, by definition, they have recovered 100 percent or went back to their original length. Even though they have all recovered completely, there are four quite different types of behavior that can be found in characteristically different materials. The reason for this is that a specimen may recover either rapidly or slowly, and also that the total amount of recovery (not the percentage) may be either large or small, depending on whether the specimen is stiff or compliant.

These four permutations lead to the four main kinds of resiliency, and these can be typified in an approximate way by quartz, rubber, wool, and uncross-linked or reduced wool. Rubber, for example, recovers almost instantly from moderate extensions, whereas wool, on the other hand, has a more de-

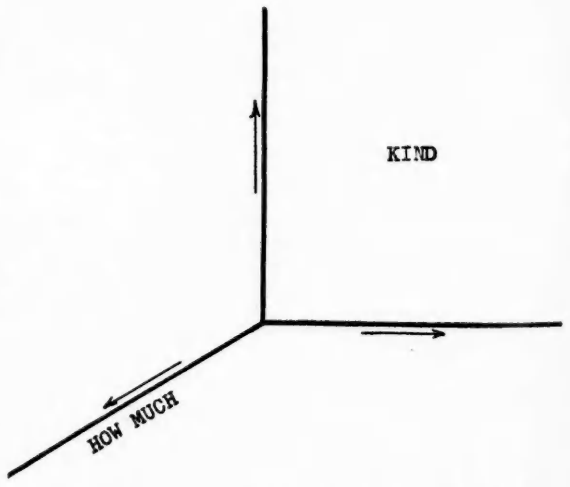

FIG. 1. *Illustration of a three-dimensional plot in which two parameters establish the kind and the third indicates the amount or extent of a complex property such as resiliency.*

layed type of recovery. This indicates that the effect of time on the stress-strain behavior is small in the one case and large in the other. An additional major difference is the fact that the modulus for rubber is relatively low compared to that for wool. It is true that wool's modulus is actually low-to-medium in comparison with the moduli of many other fibers, but it is the best example which has been found so far to illustrate the high stiffness-high delayed recovery combination. When the cross-links are broken in wool, the most significant change, according to Harris [2] and his coworkers, is a loss of stiffness or a drop in the modulus. For reduced wool the recovery from extension, while very slow, will be complete if enough time is allowed; therefore it serves to exemplify the fourth type of resiliency, as shown in the table below:

	Quartz	Rubber	Wool	Uncross-linked wool
Modulus	High	Low	High	Low
Time effect	Low	Low	High	High

These two variables, the modulus and the time effect (kind of resiliency), together with percent "final" recovery from deformation (extent of resiliency), give the three parameters for constructing resiliency maps.

The Resiliency Map

Figure 2 is a resiliency map for a number of natural and synthetic fibers studied in this laboratory. This map is intended to present only a qualitative idea of the relative positions of the various fibers, and the experimental data will be given in later papers. Furthermore, attention is focused here for the moment only on the kind of resilience, and the third or "Z" axis, which is reserved for the degree or extent of resiliency, is not used. The vertical axis shows the intrinsic stiffness or modulus of the fibers as determined from the initial slope of the stress-strain curve. A logarithmic scale is used to condense the wide range of values. The horizontal axis indicates the magnitude of the time effect, the measurement of which is discussed in more detail below.

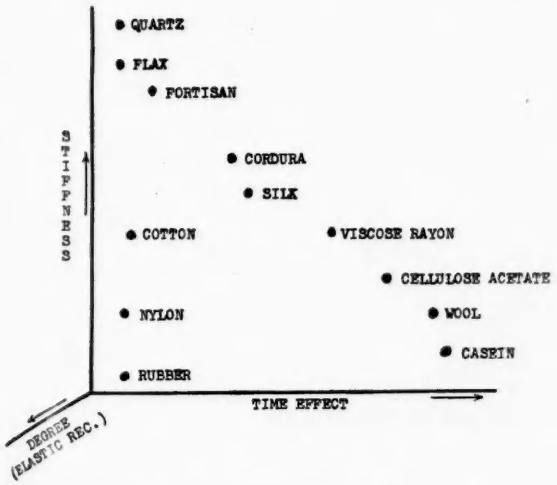

FIG. 2. *Two-dimensional map showing the relative positions of a number of materials having different kinds of resiliency.*

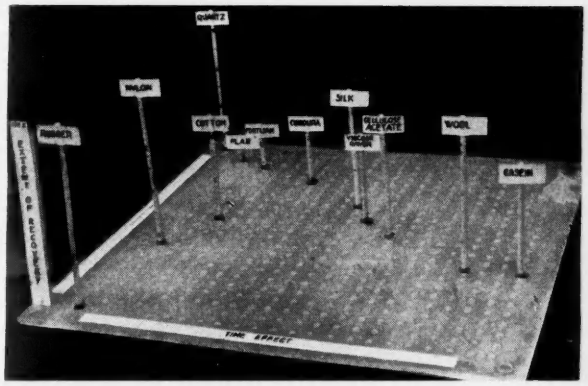

FIG. 3. *Photograph of a three-dimensional map showing the kind and extent of resiliency characteristic of various materials.*

A study of such a map will bring out a number of interesting relationships and differences among various textile yarns. It can be seen that wool and casein are indeed in a class by themeselves. Nylon stands apart, as does flax or linen. It is not surprising that general-purpose yarns such as viscose rayon are found near the center of the map. Such yarns do a good job in many types of fabric and in many applications of these fabrics, but not the best job in some special use requiring more extreme characteristics. Viscose rayon is not stiff enough, for example, to duplicate the hand and draping qualities of a linen tablecloth or compliant enough to use as the starting material for an elastic garment such as a girdle. Nor will it compete successfully with wool in certain applications for reasons that will be discussed.

It is believed that a superior wool may lie somewhere at the upper right of the map and a superior silk somewhere at the upper left. These may some day be found in the growing number of synthetic

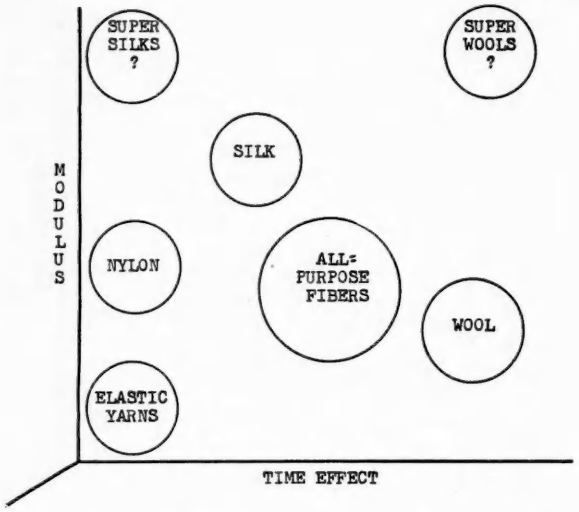

FIG. 4. *The zones of importance to textiles on the resiliency map.*

polymers. Highly oriented celluloses, such as linen or Fortisan, cannot be classed as superior silks because they fall down in extent of resiliency; hence the fact that a third axis exists must be kept in mind for all of these fibers. This can best be brought out by the construction of a three-dimensional contour map such as that in Figure 3.

The use of wet instead of dry physical-property data markedly alters the position of many fibers on these maps. In the presence of water, casein and viscose rayon suffer a marked reduction in extent of resiliency, whereas wool is improved.

In Figure 4, the approximate positions of a number of zones of importance in textiles are indicated.

The Time Effect in Stress-Strain Behavior

The time effect enters into numerous physical tests, and therefore a number of different methods have been and can be devised for measuring it. Two approaches that are being used in this laboratory can be mentioned. The first of these is shown in Figure 5, which represents the creep and creep-recovery behavior of wool and nylon as a plot of elongation versus log time.

The slope of the creep-recovery line taken at a standard load or at a standard elongation provides one measure of the time effect. Or, instead of rates, the difference in recovery measured at two times, such as at 1 minute and at 1,000 minutes later, can be used.

The "Z" axis in Figure 1, or the vertical height of the pegs in Figure 4, is reserved for the third parameter, which indicates the extent of resilience. This can be measured by the percent recovery in 24 hours from a 3-percent extension reached in a preceding creep test of 24 hours' duration. Instead of creep tests in which the yarn is gradually extended, the yarn can be immediately stretched to the desired elongation, held for 24 hours, and its recovery behavior recorded. In fact, this latter method can be used to cover any kind of deformation, such as compression, bending, torsion, and even crease tests on fabrics.

The evaluation of "permanent" deformation and "final" recovery requires an entirely arbitrary choice for the length of the recovery period. An inherent difficulty lies in the fact that most recovery curves are fairly linear when plotted against the logarithm of the time. Plastics have been found to recover continuously, although at a steadily diminishing rate, for periods up to several years. In the case of wrinkled wool fabrics tested in this laboratory, it has been found that the creases gradually disappear over a period of a day or two. Based on this and similar observations, a 1,000-minute recovery period is regarded as a reasonable choice and is believed to be comparable to that of actual textile usage. A similar period has been chosen for the duration of the original deformation, since the time effect is important during the original deformation period as well as during recovery. A woolen fabric will recover quickly from a crease test of short duration, but more slowly from

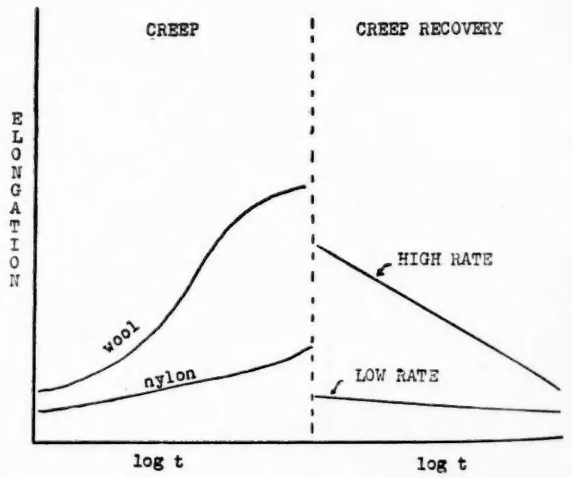

FIG. 5. *Creep curves illustrating the widely different behavior of nylon and wool with respect to the effect of time on the elongation reached under a constant load and the subsequent recovery after removal of load.*

a 24-hour test. The latter approximates what happens to a fabric when it is packed overnight in a suitcase.

For an approximate index of the time effect, use may be made of the modulus ratio M_i/M_a. In this ratio, M_i is the ordinary or initial modulus obtained from the stress-strain curve, and M_a is the average modulus out to the breaking point. M_a can be found by dividing the tenacity T by the breaking elongation E. The reason that this ratio represents a time effect can be illustrated in terms of springs and dashpots. A screen door without a spring would be a nuisance because it would not close properly; in other words, it would be nonresilient. This could be remedied by the installation of a screen-door spring. Such an arrangement is still rather a nuisance because it will slam every time little Johnny runs out the back door. So the next step is to change the kind of resilience by putting on a dashpot such as can be purchased in a hardware store. Now the system has a time effect and the resilience, although of the same high degree, is now of a different kind embodying a larger time effect.

In terms of very simple mechanical elements, these changes are shown in Figure 6.

For the simple spring, the initial and average moduli are the same and their ratio is unity. When part of the spring is restrained by a dashpot, the system is temporarily stiffened; the stress-strain curve starts out at a higher slope than would the original spring alone shown by the dotted line. However, by the time the breaking point of the yarn is approached, the dashpot becomes extended and carries less and less load. Accordingly, the stress drops back to the spring line, whose slope is approximately T/E.

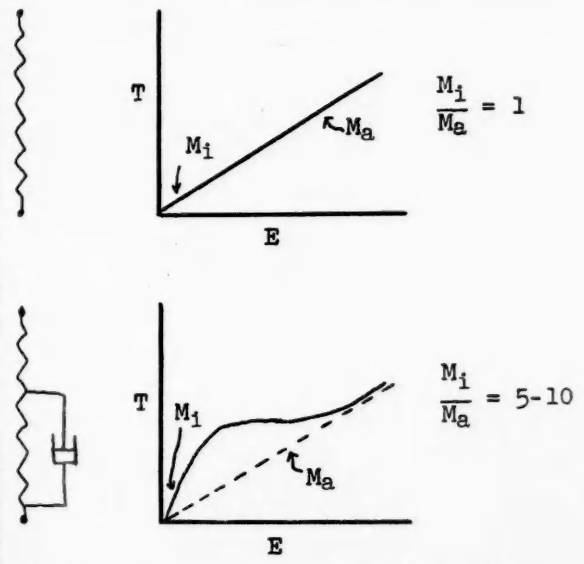

FIG. 6. *Illustration in terms of stress-strain curves of the effect of placing a dashpot in parallel with a spring and the resulting change in the ratio of the initial modulus to the average modulus.*

Instead of extending the yarn 20–30 percent in 3 minutes, as is done in an ordinary stress-strain test, a similar approximate value for the spring constant can be found by measuring the load necessary to stretch the fiber only 3 percent, but over the much longer time of 1,000 minutes. Either way gives a measure of the temporary stiffness due to the presence of a dashpot in the mechanical analogues used for describing fiber behavior.

Work absorbed by a dashpot cannot be returned; in other words, as a dashpot operates, the work recovery is lowered. This would indicate that wool has a low work recovery, although quite the opposite has often been said. The trouble was that not all of the data were considered. Wool has a high work recovery for small elongations before the dashpot gets under way. It has a lower work recovery for higher strains, as shown in the data below, in which it is compared with nylon:

	Work Recovery (%)			
Extension (%)	0.5	1	2	5
Wool	94	87	62	29
Nylon	82	82	82	75

The work-recovery test has been a useful, but sometimes misleading, criterion of resiliency. A work recovery of 100 percent is sufficient to indicate that the recovery from strain is also perfect. But a work recovery as low as 80 percent or less can often be accompanied by complete recovery or freedom from permanent deformation, as is true for both nylon and wool at certain elongations.

The Change of Modulus with Time and Strain

The need not only for a high degree of resilience but also for the right kind of resilience is particularly well illustrated by fabrics of worsted-type construction. A spring-like or rubber-like elasticity is not enough. An additional characteristic possessed by wool is its diminishing modulus. The stiffness of wool decreases rapidly with the load and elongation and with the duration of the load. It is in this connection that the "time effect" becomes useful. As a

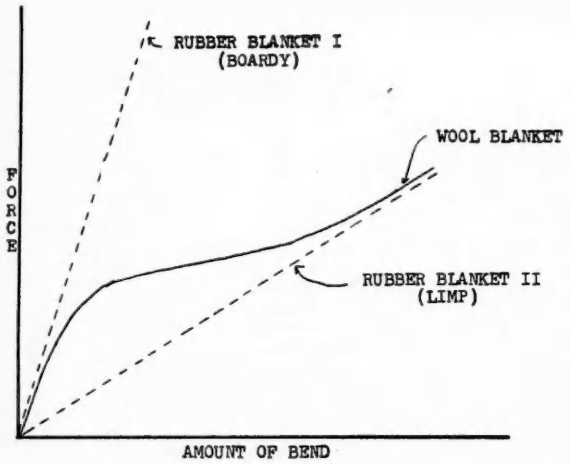

FIG. 7. *Comparison of the bending behavior of a wool blanket with rubber blankets of two different thicknesses.*

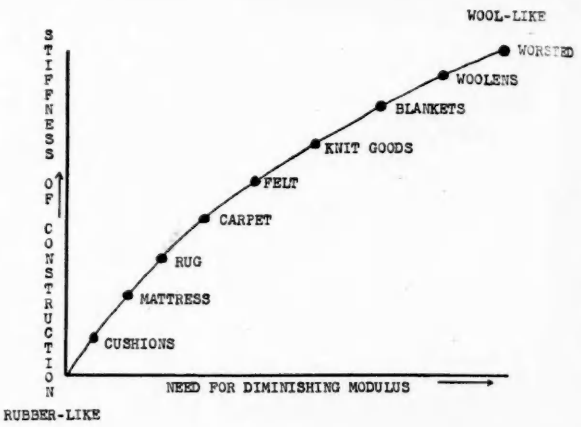

FIG. 8. *A series of applications arranged according to stiffness indicating how the requirements vary from a simple rubberlike modulus at the start to the more complex diminishing modulus which is characteristic of wool and which is particularly necessary in the denser, harder fabric constructions, such as worsteds.*

result, wool has a dual nature. It is fairly stiff and spring-like for short loading times and low strains, but it becomes increasingly compliant at longer loading times and higher strains. This dual nature underlies the fact that a good woolen fabric can be described as being crisp and yet soft. This is just the opposite of the so-called "dead" fabrics, which are either too limp or too harsh and boardy, depending on the weight of the material used. This is illustrated in Figure 7.

It is possible to imagine two blankets, one of wool, the other of rubber, which by the proper choice of weights of these materials have been so made that they both offer the same resistance to small deformations. However, at higher deformations the rubber blanket would turn out to be perhaps twice as stiff as the woolen one; as a result it would be considered harsh and boardy. The woolen one could be folded into a neat bundle; the rubber one would resist this operation and might even partially unfold as soon as the hands of the folder were removed. A thinner rubber blanket, such as No. II in Figure 7, constructed to duplicate the stiffness of the woolen one at higher strains, would be too limp initially.

The versatility of wool, due to its diminishing modulus, is further illustrated in Figure 8. In certain jobs rubber and wool can do equally well. These are applications in which the forces accompanying deformation are relatively small and only the initial spring-like part of the stress-strain curve is involved. Either sponge rubber or wool batting will make a resilient cushion or mattress. Proceeding up the series towards applications involving greater stiffness, the need for the diminishing modulus which is characteristic of wool—and to a lesser extent characteristic of certain other fibers, such as cellulose acetate and casein—becomes increasingly apparent.

The stiffness of a fiber can be calculated from the amount of load necessary to bring about a certain elongation, but it is meaningless to do this without specifying the time involved. Most fibers act as non-Hookean springs; therefore, it is also necessary to specify the elongation at which the calculation is made because the stiffness varies with the length to which the fiber is stretched. These two effects are illustrated by the table below, in which the stiffness in grams per denier per 100 percent elongation has been calculated for a wool fiber from creep data.

Stiffness of a Wool Fiber at Various Elongations and Loading Times

Time (min.)	1%	2%	5%
0.1	49	37	18
10	40	32	16
1000	31	26	14

The modulus of wool, determined by the velocity of sound and involving much shorter times (1×10^{-6} minutes) and elongations, is 56.

Creep data for nylon show a similar but smaller drop of stiffness with time. More striking, however, is the fact that the stiffness of nylon increases with elongation. This is true whether it is calculated by

L/E (the secant modulus) or by dL/dE (the differential modulus).

This effect can also be demonstrated by the velocity-of-sound method; nylon and rubber increase and wool decreases in stiffness with elongation. It is reasonable to expect the stiffness to increase as the kinks in the molecular chains are pulled out and no longer contribute to ease of extension. For wool, on the other hand, the sonic modulus measured at various elongations at first decreases, reaches a minimum, and then slowly increases. This is in accord with the view that the folds or kinks in the wool chain double back upon themselves and that these loops are held in a closed position by hydrogen bonds. These attractive forces are magnet-like (rather than spring-like) in that they vary in strength inversely with the distance apart or with the elongation of the fiber as a whole. These forces undoubtedly account for much of the initial crispness and lively recovery behavior of wool. It is one of the few fibers which shows (on wet tests) a concave upward part of the hysteresis loop during recovery, indicating its ability to snap back instead of crawling back to its original length.

The Relation of Modulus and Work Absorbed

In his 1944 Edgar Marburg Lecture, Harold De Witt Smith [3] defined resilience as the ability of a material to absorb work without suffering permanent deformation. If we examine this definition closely, we find that it can be amplified along the lines of the generalized concept presented here. The problem of extending his definition lies in two words. One is the word "permanent." This has led us to ask the question, "Measured when?" In other words, we must bring time into the definition. The second is the word "work." Work is defined as force times distance, or the area under the initial part of the stress-strain curve, which is approximately one-half of the product of load and elongation:

$$W = 1/2\ LE. \qquad (1)$$

There are, however, two other important ways of representing this area. Using the slope of the stress-strain line, $M = L/E$ = slope = stiffness, two additional equations can be obtained:

$$W = 1/2\ ME^2 \qquad (2)$$

and

$$W = 1/2\ L^2/M. \qquad (3)$$

Thus it can be seen that the amount of work that can be put into a textile system is governed by the stiffness. In those problems involving constant strain or elongation equation (2) applies, and the work absorbed is directly proportional to the stiffness.

In the case of constant-load problems (equation 3), the stiffest material absorbs the least work and M appears in the denominator. This makes it readily apparent why a quartz plate, with a high stiffness, can absorb only a little work under a specified load and would be a poor carpet material. Sponge rubber or a wool-pile carpet each has a low stiffness and can therefore produce the effect desired in a luxurious carpet. It has been reported [4] that various materials can be rated for carpet use by the work of compression under constant load (rather than by the work recovery). Actually, the reciprocal of the modulus will rate such materials in the same order if the stress-strain curves are nearly linear.

Resilience and Resiliency

The presence of a stiffness parameter in the generalized concept makes it possible to attack with some hope of success an otherwise very troublesome aspect of the resilience problem. This complication is the so-called "form factor," which in textile applications arises from such variables as the shape and size of yarns and filaments, the twist, the crimp, and the fabric construction, etc. The form factor can be illustrated by comparing the properties of a glass plate and a batting of spun glass fibers. The intrinsic stiffness or modulus is the same in both cases, but the actual stiffness is quite different. This is similar to numerous other instances which require a careful distinction between inherent or specific properties and dimensional or "total" properties [5] depending not only on the substance, but on its size and shape. One speaks of the conductivity of iron and of the conductance of an iron wire of a certain size.

It is proposed, therefore, that *resiliency* (and modulus) be used in referring to a substance, such as glass, nylon, rubber, wool, etc., and that *resilience* (and stiffness) be used in referring to an entity, such as a woolen fabric, a nylon yarn, or a steel spring, in each case one of specified dimensions and construction. In the proper physical form, quartz might make a resilient cushion material. Between a solid plate and a fibrous batting of glass there is, therefore, a difference in resilience, but not in resiliency. It is

entirely possible that two different fabrics made from the same fiber may vary widely in stiffness and resilience [6]. In addition to the obvious effect which the form factor exerts on stiffness, it can also influence other stress-strain-time relationships. When crimp is present in a fiber, for example, the apparent stretch and the real stretch may be different. This may greatly minimize the amount of semi-permanent deformation under a given extension and alter the recovery behavior both in extent and in time dependence.

Summary

In summary, the resiliency of a material is a complex stress-strain-time property described by the combination of three parameters. Two of them are relatively simple—the modulus and the extent of recovery from strain. But both of these quantities vary with time and strain, and the description of this variation is the function of the remaining parameter. In tabular form this is:

Axis	Quantity measured
Y	Initial modulus: M_i
Z	Extent of recovery from strain: R
X	Change of modulus and recovery with time and strain: ΔM and ΔR

For a definition, the following is proposed: Resiliency is a stress-strain-time property of a material, characterizing the completeness of recovery from deformation and varying in kind with the modulus of elasticity and the rate of recovery.

Corollaries of this definition are:

1. Four main types of resiliency arise from the four permutations of high and low moduli with high and low rates of recovery.

2. The extent of resiliency is the degree of recovery in a specified time.

3. Resiliency is a specific property of a substance; resilience is a total property depending not only on the material but on the size and shape.

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Improvements in the Acid-Hydrolysis Method of Determining the Crystallinity of Cellulose*

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Abstract

In a study of the acid-hydrolysis method [5] for the determination of the degree of crystallinity of cellulose fibers, a technique has been developed for the solvent removal from the hydrolysis residues of all but traces of the humic substances which are formed from the soluble hydrolysis products and which interfere with the crystallinity determination.

Although ammonium hydroxide and pyridine were effective solvents, hot 50-percent aqueous monoethanolamine was found to be the most practicable means of removing these humic substances. Its effectiveness was demonstrated (*a*) by showing that the weights of the extracted residues were the same as the weights of residues corrected by computations based on data from experiments with glucose; (*b*) by varying the liquid-solid ratio during hydrolysis and showing the uniformity of the weights of the resulting ethanolamine-extracted residues; and (*c*) by obtaining equal extrapolated values for degree of crystallinity from parallel extracted and unextracted but mathematically corrected hydrolysis series.

Data are presented to show that preliminary extraction with either 1-percent sodium hydroxide or ethanolamine to remove the noncellulosic constituents does not change the crystallinity values of samples of cotton and cotton linters.

A revised crystallinity procedure is described which incorporates the improvements described above.

IN THE PROCEDURE for determining "degree of crystallinity" of cellulose fibers described by Philipp, Nelson, and Ziifle [5], the rate of hydrolysis of cellulose is determined at elevated temperatures in the presence of hydrochloric acid.

A problem which has complicated the experimental application of this acid-hydrolysis technique is the contamination of the cellulose residues by insoluble humic substances which are formed from the glucose produced by cellulose hydrolysis and possibly from other soluble hydrolysis products. Another problem, that of the effect of preliminary sample purification, arose when it was desired to make crystallinity determinations on cellulose samples containing considerable quantities of noncellulosic materials.

This paper presents (*a*) a method for separating and removing the humic substances from the cellulose residues; (*b*) a study of the effect of a preliminary purification of samples on the crystallinity values obtained; and (*c*) a revision of the previously proposed procedure for crystallinity determinations, including several suggestions on laboratory technique.

Removal of Humic Substances

Problem of the Humic-Substance Correction

In the acid-hydrolysis procedure, the "degree of crystallinity" (defined as the amount of crystalline cellulose expressed as a decimal fraction of total cellulose) is obtained by extrapolation of data defining the hydrolysis rate of the crystalline fraction, which conforms to the equation for a first-order reaction:

$$\ln c = \ln c_o - k_c t,$$

where c = amount of unhydrolyzed crystalline cellulose at time t, c_o = original amount of crystalline cellulose, and k_c = hydrolysis rate for crystalline cellulose. The extrapolation to obtain c_o may be performed either graphically or by calculation.

Experimental determination of values of c (the unhydrolyzed residue at various times) has been complicated by the deposition on the hydrolysis resi-

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Another paper on the crystallinity of cellulose, by the same authors, appears on page 155.

† One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

TABLE I. HYDROCELLULOSE RESIDUES REMAINING AFTER VARIOUS EXTRACTIVE TREATMENTS

Treatment	1-hour hydrocellulose (%)	20-hour hydrocellulose (%)
Control, wet with hot water	100.0*	100.0†
Boiled 30 min. with water	100.3*	99.8†
Boiled 5 min. with 50-% ethanolamine	99.8	97.0
Boiled 30 min. with 50-% ethanolamine	99.0	94.2
Boiled 30 min. with pyridine	99.8	100.4

* Negligible quantity (less than 0.1 percent) of humic substances present.

† Corrected for presence of humic substances by subtraction of "humic correction" (calculated from independent data) from original weight.

dues of small quantities of insoluble humic substances which are formed from the soluble hydrolysis products. A previous paper [5] describes in detail a method for making a stepwise mathematical correction for the presence of these humic substances. This method, however, requires not only considerable calculation but also more regular and frequent observations than are necessary merely for the calculation of degree of crystallinity; therefore, the present study was undertaken to develop a technique for chemically removing the humic substances from the residues before weighing.

Selection of Solvents

In the preliminary experiments to find a solvent which would remove the humic substances without affecting the hydrocellulose in the residues, various common laboratory solvents were tried. It immediately became apparent that alkaline solvents such as pyridine, ammonium hydroxide, or monoethanolamine (hereafter referred to as "ethanolamine") would dissolve the humic substances.

Although sodium hydroxide solutions will dissolve the humic substances, they were not studied, as it is well known, as shown by Davidson [2], for example, that they will dissolve acid-degraded cellulose to an extent dependent upon the amount of previous degradation. Pyridine and ammonium hydroxide solution appeared to give satisfactory results but they are undesirable for routine use because of their volatility and unpleasant odors. Ethanolamine, on the other hand, does not have these undesirable properties, and, since it is an effective solvent at the same time, it seemed the logical choice for further study.

Pure ethanolamine has a rather high viscosity (21.2 centipoises at 25°C compared to about 0.9 for water) which makes its passage through "fine"-porosity fritted crucibles quite slow. Both 50-percent and 25-percent aqueous solutions (by volume) are sufficiently less viscous (viscosities 6.89 and 2.20 centipoises, respectively, at 25°C) to permit rapid filtration. All three concentrations (100-percent, 50-percent, and 25-percent) were used in preliminary experiments and it was found that the diluted solvent performed satisfactorily. For the standardized procedure it appeared that the 50-percent solution would be preferable since it represents a compromise between lowered viscosity and reduced concentration of the solvent.

Solubility of Cellulose in 50-Percent Ethanolamine

To determine whether boiling 50-percent ethanolamine will dissolve appreciable quantities of acid-degraded cellulose, which would interfere with its use as a solvent for the humic substances, samples were prepared by hydrolyzing cotton linters with 6*N* hydrochloric acid at 100°C. One batch was heated for 1 hour and another batch for 20 hours. The acid was removed by filtration and the residues were washed well with water and allowed to air-dry. Small samples were weighed out and extracted with 50-percent ethanolamine for either 5 or 30 minutes. For comparison, parallel samples were extracted for 30 minutes with water or with pyridine and a control sample was merely wetted with water. On the basis of appearance as well as on that of previous data, it was known that the 1-hour hydrocellulose was essentially free of humic substances; therefore, for this series the weight of the residue that was wet with water only and redried could be used directly for the "control." On the other hand, the 20-hour hydrocellulose was contaminated with a considerable quantity of humic substances. To obtain a "control" value for the hydrocellulose content of this sample a correction calculated from independent data was applied.

The results of these treatments are shown in Table I. It can be seen that boiling 50-percent ethanolamine dissolves a small percentage of acid-degraded cellulose, but for a 5-minute extraction period the quantity is negligible for moderately degraded material, and not excessive for even the very highly degraded 20-hour hydrocellulose. Since the 20-hour hydrolysis residue is only about 25 percent

TABLE II. CONTAMINATED-RESIDUE PERCENTAGES AND PERCENTAGES CORRECTED BY COMPUTATION AND BY ETHANOLAMINE EXTRACTION

Duration of hydrolysis (hr.)	Contaminated residues (%)	Residues corrected by computation (%)	Residues extracted with ethanolamine (%)
	Residues from cotton linters *		
2	75.1	75.1	74.9
3	69.9	69.9	70.0
4	65.0	64.9	64.3
5	60.8	60.7	60.6
6	57.5	57.3	56.4
7	54.8	54.5	54.3
8	50.7	50.3	49.6
16	31.3	29.9	30.2
20	24.4	22.4	23.4
24	21.3	18.8	18.7
30	14.9	11.8	11.4
	Residues from cotton tire cord †		
5	51.9	51.7	51.9‡
7	44.9	44.4	44.5‡
16	23.2	21.4	21.8
24	14.3	11.3	11.6
30	10.2	6.6	7.2

* Averages of duplicate determinations.
† Averages of quadruplicate determinations except where otherwise noted.
‡ Averages of triplicate determinations.

of the original cellulose sample, the 3-percent loss by a 5-minute ethanolamine extraction, when recalculated on the basis of the original cellulose sample, becomes only 0.75 percent, which is about the magnitude of the experimental error. The progressive loss of hydrocellulose on heating with ethanolamine for the longer time indicates that the extraction period should be limited to 5 minutes.

Extent of Humic-Substance Removal

To determine whether 50-percent ethanolamine was removing the humic substances adequately, the three following tests were made.

First, paired hydrolysis series were run using 6*N* hydrochloric acid at 100°C for various time intervals. In one series of each pair the experimental residue percentages were corrected for the presence of the humic substances by application of calculated corrections, as explained before. The residues of the other series were extracted with ethanolamine to remove the humic substances. The corresponding residue percentages should, of course, be equal. Results from two different sets of parallel series are given in Table II. The closer agreement between comparable values in the second set (cotton tire cord) can be attributed to the greater number of replications entering into each average.

TABLE III. RESIDUE PERCENTAGES OBTAINED WITH AND WITHOUT 50-PERCENT ETHANOLAMINE EXTRACTION AFTER HYDROLYSIS WITH DIFFERENT LIQUID-SOLID RATIOS

Liquid-solid ratio	Extracted residue (%)	Unextracted residue (%)
500:1	17.6	19.6
250:1	17.7	20.3
100:1	18.1	22.8

A second test of the effectiveness of the extraction technique involved a comparison of residue percentages obtained by varying the liquid-solid ratio. Since the heterogeneous hydrolysis of crystalline cellulose appears to follow a first-order reaction, the rate should be independent of the initial concentration of the cellulose and differences in liquid-solid ratio should produce no difference in percentage of cellulose hydrolyzed in a given time. This was previously demonstrated [5] for samples hydrolyzed for 1-hour intervals at ratios of 100:1, 250:1, and 1,000:1. However, after a long period of hydrolysis, the uncorrected residue percentages from such a series of ratios would no longer be equal, owing to the presence of varying quantities of humic substances. The formation of these substances, as previously explained, follows approximately a second-order reaction and is therefore strongly influenced by initial concentration of reactants.

It was postulated that if ethanolamine extracts the humic substances completely then different liquid-solid ratios should give the same percentage of extracted residue after equal hydrolysis periods, since the solvent should remove the humic substances which were responsible for the variations in residue percentages. The results of such an experiment with samples of cotton linters, hydrolyzed with 6*N* hydrochloric acid at 100°C for 24 hours, are given in Table III. As will be seen, the values for residues extracted with ethanolamine are equal within experimental error, whereas the values for unextracted residues increase with decreasing liquid-solid ratio —that is, as the concentration of glucose formed in hydrolysis increases. This constitutes additional proof that the extraction technique is adequate.

The third test of effectiveness, and the most important requirement for the use of the 50-percent ethanolamine extraction technique, is that it will give

TABLE IV. CRYSTALLINITY VALUES OBTAINED WITH AND WITHOUT USE OF SOLVENT FOR HUMIC SUBSTANCES

Set No.	Material	Solvent	Degree of crystallinity
	Cotton linters A	None *	0.89
	Cotton linters A	Ethanolamine, 100%	0.89
I	Cotton linters A	Ethanolamine, 50%	0.88
	Cotton linters A	Ethanolamine, 25%	0.88
	Cotton linters A	Pyridine	0.88
II	Cotton linters B	None *	0.81
	Cotton linters B	Ethanolamine, 50%	0.80
III	Cotton tire cord	None *	0.88
	Cotton tire cord	Ethanolamine, 50%	0.86
	Cordura rayon	None *	0.59
IV	Cordura rayon	Ethanolamine, 50%	0.60
	Cordura rayon	Ammonium hydroxide	0.59

* Calculated humic corrections applied to residue weights.

the same value for degree of crystallinity when applied to various types of samples as that derived from unextracted series to which the calculated correction for humic substances has been applied. Four sets of parallel series were run using two different samples of cotton linters, a cotton from tire cord and a viscose rayon known as Cordura. The cotton linters A was a stock lot of low-viscosity purified linters. The cotton linters B was the same material which had been subjected to a previous treatment that reduced the degree of crystallinity. The tire cord was made from Stoneville 2B variety of cotton, and had not been previously purified. The Cordura rayon was a tire cord-type rayon. The results are presented in Table IV. The good agreement between values for degree of crystallinity obtained by different methods shows that humic-substance extraction with ethanolamine is a workable technique.

Sample-Purification Treatments

Samples Requiring Purification

Much of the preliminary work on the development of the acid-hydrolysis method for determining degree of crystallinity was done on stock lots of purified cotton linters, viscose rayons, and the like [5] which, because of the previous treatment, contained only minute amounts of noncellulosic constituents. The acid-hydrolysis method was later applied to a number of raw cottons. These contain from 4 percent to 12 percent of constituents other than cellulose, such as proteins, pectic substances, ash, and wax, depending on growth conditions and variety [3]. As long as the percentage of such materials was low, it did not appear that the noncellulosic constituents interfered. However, when crystallinity determinations were made on samples such as very immature cottons and partially degummed ramie, containing large amounts of noncellulosic materials, erratic results were obtained. Not only was it uncertain whether or not these noncellulosic constituents completely dissolved during hydrolysis, but also the heterogeneous nature of such materials made adequate sampling difficult. A preliminary purification treatment was indicated, provided that it could be done without changing the crystallinity values.

Choice and Examination of Purification Treatment

Since alkali extraction is a conventional as well as one of the most effective means of removing nitrogenous constituents and pectic substances [4, 6], experiments were undertaken to determine the effect of alkali extraction upon the subsequently determined degree of crystallinity.

The samples used were (*a*) cotton linters, (*b*) a normal mature cotton, (*c*) a very immature cotton, and (*d*) Arkansas green-lint cotton, which is not only very immature but also contains 12 to 15 percent of wax [1]. All samples except the linters were first extracted for 2 to 3 hours with 95-percent ethanol in a Soxhlet apparatus at a temperature of 60°C or higher to remove the waxy substances. The extracted samples were dried in air at room temperature, ground through a 2-mm. sieve in a large Wiley mill, and divided into three portions. One 10-g. portion was heated for 2 hours with approximately 500 ml. of boiling 1-percent sodium hydroxide solution; a second 10-g. portion was heated for 2 hours with approximately 400 ml. of boiling ethanolamine (B.P. 171°C); and the third portion, serving as a control, was boiled for 2 hours in distilled water. The sodium hydroxide-extracted samples were washed well with hot water, "soured" with dilute acetic acid, and washed with hot water until neutral. The ethanolamine-extracted samples were washed until neutral with hot water only. The excess water was removed by suction and the moist cotton was spread to dry at room temperature. Crystallinity determinations by the acid-hydrolysis technique were applied to portions of each of the samples as well as to the unpurified materials and gave the results listed in Table V. The standard errors were calculated but they are not

shown in the table; they ranged from 0.005 to 0.016, with an average value of 0.01.

In general, agreement between controls and extracted samples is good. However, two values appear to be somewhat out of line. One, the degree of crystallinity of unextracted immature cotton, is significantly different, statistically, from the three values obtained after alcohol-and-water- or alkali extraction. Since alcohol extraction is a very mild treatment, it seems unlikely that it would affect the crystalline-amorphous structure; it is more probable that the crystallinity value for the unextracted material is too low, especially in view of the excellent agreement between the water- and alkali-extracted samples. This low value may be due to interference of the noncellulosic constituents during the crystallinity determination. The other discrepancy is the rather high value for green-lint cotton after extraction with alcohol and ethanolamine. In this case, however, the difference between the extreme values (0.86 and 0.81) was found to be not significant statistically, owing to the rather large standard errors of the values in this series. It was therefore concluded that a preliminary alkali extraction does not appreciably affect the value obtained for degree of crystallinity by the acid-hydrolysis method.

TABLE V. DEGREE OF CRYSTALLINITY OF DIFFERENT COTTON SAMPLES BEFORE AND AFTER CERTAIN PURIFICATION TREATMENTS

Material	Not extracted	Hot alcohol extraction followed by extraction with: Water (control)	Sodium hydroxide	Ethanolamine
Cotton linters	0.88	—	0.89	0.89
Normal cotton	0.84	0.84	0.86	0.86
Immature cotton	0.81	0.86	0.86	0.87
Green-lint cotton	0.81	0.81	0.83	0.86

Recommended Procedure

As a result of the present studies, as well as other studies to be reported elsewhere, the following procedure is recommended for the crystallinity measurements:

The sample, either raw or purified by a 2-hour extraction with boiling 1-percent sodium hydroxide or boiling 100-percent ethanolamine, is cut into small pieces and thoroughly mixed. If the nature of the material requires that the sample be ground, grinding should be done in a Wiley-type mill with 2-mm. or coarser sieve. Fine grinding is not recommended, since, as will be shown in a subsequent report, the crystallite structure is affected. As many 0.2-g. samples as required are weighed out into 25 × 200-mm. test tubes which are provided with permanently engraved rings at 50-ml. capacity, as described in a previous report [5]. Along with these, samples for moisture and cellulose determinations are weighed out. These serve for the subsequent expression of the results on the basis of the cellulose content.

The samples in the large test tubes are covered with boiling hydrochloric acid of appropriate normality up to the 50-ml. ring, according to a staggered time scale, and are placed in a rack in a constant temperature bath at 100°C. The normality of the acid will ordinarily be 6 for undegraded celluloses and 4 for regenerated celluloses. Although the normality employed does not directly affect the crystallinity value found, it does greatly affect the hydrolysis rate, and too great a rate reduces the accuracy with which the residue weights can be determined, and, in turn, the confidence limits of the extrapolated crystallinity value. It is advisable to add approximately half of the boiling acid to the sample and then to shake the test tube vigorously in order to wet the sample thoroughly; otherwise, portions of the sample frequently float on top of the acid, giving rise to erratic residue weights. The remainder of the acid is now added and the tube is closed with a ground-glass cover, the joint being sealed with a heat-resistant lubricant to prevent evaporation.

Ordinarily, duplicate or triplicate samples are started for each hydrolysis interval, and these are staggered in time so that the hydrolysis can be stopped at the proper moment by filtration and washing.

When the predetermined hydrolysis period has elapsed, the tube is quickly removed from the bath and the contents are filtered on a fine-porosity fritted glass crucible. It is important at this point, as well as in the subsequent extraction of the humic substances and washing of the residue, that the latter never become dry on the filter. Failure to observe this precaution usually results in clogging of the filter pores, which greatly decreases the rate of filtration.

When a small quantity of water remains after washing out the acid, the residue is transferred, with the aid of a stream of hot 50-percent ethanolamine from a wash bottle, to a small (150-ml.) beaker. The total volume used is about 30 ml. The beaker

is covered and the residue is digested on a hot plate for 5 minutes. The temperature of the hot plate is adjusted so that the temperature of the solvent is maintained near the boiling point (110°–115°C) but the solution does not "bump." The material is refiltered through the same crucible, washed well with hot water, and dried in an oven at 105°C.

The residues are computed as percentages of original dry weight if purified samples were used, or as percentages of the cellulose content by separate cellulose determination if raw celluloses were employed. The residue percentages are now plotted on the logarithmic scale of semilogarithmic paper against time on the linear scale, and the straight-line portion represented by the longest hydrolysis periods is extrapolated to zero time. Alternatively, the residue percentages which after preliminary plotting are found to fall on the straight-line portion of the curve may be treated mathematically by finding the product-moment regression line for equation (1) and solving for c_o—that is, c at zero time.

Summary and Conclusions

The removal of the humic substances by solution in 50-percent aqueous ethanolamine has been shown to be a satisfactory method of eliminating these substances from the residues in the acid-hydrolysis procedure for determination of degree of crystallinity. The amount of hydrocellulose that goes into solution during a 5-minute extraction period is negligible. The efficacy of the solvent in removing humic substances has been demonstrated (*a*) by comparing hydrolysis residue percentages for parallel series, (*b*) by comparing residue percentages from hydrolyses performed with different liquid-solid ratios, and (*c*) by showing that the same extrapolated degree of crystallinity is obtained either by mathematical correction or by solvent removal of humic substances.

A preliminary purification of cellulose samples by 2-hour extraction with either boiling 1-percent sodium hydroxide or boiling monoethanolamine has been studied and appears to have no significant effect on subsequently determined crystallinity values. Such a preliminary extraction may be made a part of the routine procedure if desirable; or it may be applied only when the noncellulosic content of the sample is rather high.

On the basis of these results, a revised recommended procedure for crystallinity determinations is presented.

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Effect of Grinding on the Crystallinity of Cellulose Fibers, as Indicated by the Acid-Hydrolysis and Other Techniques*

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Abstract

A study was made of an effect previously noted, that grinding cotton fibers in a Wiley mill causes a reduction in degree of crystallinity as determined by the acid-hydrolysis method. It is shown on samples of purified linters and cut cotton, ground under conditions of increasing severity, that even mild grinding—for example, through a 2-mm. sieve—causes a detectable reduction in indicated degree of crystallinity, and that as the severity of grinding increases the degree of crystallinity drops correspondingly. This drop is not caused by passage of more of the finely ground particles from the more severely ground samples through the filters. When the ground samples were moistened with water and allowed to dry, a portion of the lost crystallinity was regained.

The acid-hydrolysis results are supported by accessibility, heat of wetting, and moisture adsorption measurements, which, with few exceptions, indicate increasingly larger amounts of amorphous material in the samples receiving the more severe grinding.

While the x-ray diffraction patterns did not reveal any effect of the most severe grinding, it is assumed that the changes were not sufficient to permit their detection by this means.

Some implications of the results on the interpretation of cellulose structure are discussed.

DURING the development of the acid-hydrolysis technique for determination of degree of crystallinity of cellulose fibers [15] it was noted that cutting in a Wiley mill to pass a 20-mesh sieve apparently caused a reduction in the indicated value for degree of crystallinity. Because at that time the interest was primarily in the development of the acid-hydrolysis method rather than in a study of the effect of grinding, all samples to be hydrolyzed were left in the natural state or were cut in short pieces with scissors in order to avoid complications due to grinding. Since, however, any appreciable effect of grinding would be a serious handicap to a generally applicable method, it was deemed desirable to investigate this apparent reduction in crystallinity.

Previous Grinding Studies

An examination of the literature showed that a number of workers have made studies which indicate that certain grinding procedures (which for the most part are considerably different from that employed in the present study) may affect the crystalline structure of cellulose fibers. Hess, Steurer, and Fromm [7], Husemann and Carnap [8], and Hermans [4, 5] found that cellulosic materials ground in a "vibratory" ball mill were broken down into greatly deformed fragments, with or without fibrillation.

X-ray investigations by Hess and coworkers [7], principally on wood pulp and ramie, ground in the "vibratory" ball mill, showed that even after 1 hour of grinding the cellulose crystal interferences could no longer be recognized in the x-ray diffraction pattern. Treatment of the ground material with water resulted in partial recrystallization, to give the x-ray pattern of hydrate cellulose. Hermans and Weidinger [6] confirmed this finding for ramie, wood pulp, and viscose rayon (that is, for both native and hydrate cellulose).

Hess and coworkers [7] found also that a large decrease in viscosity took place at the beginning of dry grinding. For instance, the viscosity of a cuprammonium solution of fir pulp decreased to one-half after 2 hours of grinding. Their viscosity

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studies were complicated by the presence of porcelain dust from the ball mill, but after application of suitable corrections the marked changes in viscosity were still found. They believed that the change in viscosity was especially significant, since it indicated a shortening of the cellulose chains, thus implying the breaking of primary valence bonds. However, since the heat of activation of this reaction was found to be practically zero, these investigators concluded that the molecular degradation must be due to the mechanical grinding action, and not to oxidative or hydrolytic influences.

Saeman [16] noted a decrease in extrapolated values for potential sugars in wood samples ground and separated by sieving into different particle-size classes. Ingersoll [9] found that cutting rayon samples in preparation for obtaining x-ray diagrams decreased the degree of lateral order (that is, crystallinity) as did also crushing the filaments by rolling.

The literature thus discloses considerable evidence of the destruction of crystal structure by grinding.

Experimental Procedure

Two sources of cellulose were employed in the experiments reported here. Sample Co-1150, hereafter referred to as "linters," represented a commercially purified batch of cotton linters having an estimated degree of polymerization of 1,225. It had been purified by heating with dilute caustic solution under pressure, followed by a bleach. Sample Co-1151, hereafter referred to as "cut" cotton, was prepared from cotton cut in a commercial machine to simulate linters and purified by the same process as the other sample. It had an estimated degree of polymerization of 860. Both samples were white and contained only minimal amounts of noncellulosic impurities.

Wiley mills were used in preparing the different ground products. A large portion of the stock sample of cotton linters was ground in a small Wiley mill with a 20-mesh sieve in place below the cutting chamber of the mill. This rather finely ground material was divided into four parts, three of which received further treatment. One part was put through the Wiley mill again, with the same 20-mesh sieve in place. A second part was reground through an 80-mesh sieve, and a third part was moistened with water and dried at room temperature. Another portion of the stock sample was ground in a large Wiley mill with a 2-mm. sieve in place below the rotor. This presumably coarsely ground material was then divided into three parts. One part received no further treatment; a second part was soaked in water and dried at room temperature; the third part was beaten with water in a Waring Blendor until the particles, when viewed under the microscope, showed signs of fibrillation, and then was dried at room temperature.

One portion of the cut cotton was ground in the small Wiley mill through a 20-mesh sieve, and another portion was ground three times through an 80-mesh sive. Part of the latter very finely ground material was boiled for 5 minutes in distilled water and allowed to dry at room temperature.

Approximately 10-g. portions of the different samples were placed in the 40-mesh basket of a nest of standard sieves and agitated for 30 minutes. The percentages of particles by weight remaining on the 40-, 60-, 80-, 120-, and 200-mesh sieves were determined. Because of the tendency of the particles to cling together, in some cases the separation was incomplete. Also, owing to the small diameter of the fibers, obviously it was possible for a portion of the particles which were longer than the nominal sieve opening to pass through.

Degree of crystallinity was determined with 6*N* hydrochloric acid according to the procedure described in a previous publication [15], the humic correction being made by solvent removal according to a recent modification [12].

The "accessibility" of certain of the samples was determined by the Conrad and Scroggie [1] modification of the Nickerson hydrolysis-catalytic oxidation method [13, 14]. The accessibility values have been calculated both by the "method of slopes" and by the "method of heights," and the results averaged, as recommended by Conrad and Scroggie [1].

Heat of wetting was determined on oven-dry samples, in a simple calorimeter, by measuring the increase in temperature of a large excess of water into which approximately 2 to 4 g. of the sample was quickly stirred. Since x-ray studies have shown that water does not penetrate into the crystalline part, it was assumed that the heat evolved was due to the adsorption of water by the amorphous fraction and by the surface of the crystallites.

Data on moisture regain were obtained by exposing samples which had been previously dried overnight in a vacuum oven at 80°C to atmospheres of 20, 65, 79, or 90 percent relative humidity at a temperature of 21°C and expressing the increase in

TABLE I. Percentages of Ground Cellulose Samples Retained on Certain Standard Mesh Sieves after Being Shaken for 30 Minutes in a Standard Sieve Shaker

Nominal width of openings (mm.)	Cotton linters					Cut cotton		
				Wiley mill sieve size				
	Not gr.* (%)	2-mm. (%)	20-mesh (%)	20- and 20-mesh (%)	20- and 80-mesh (%)	Not gr.* (%)	20-mesh (%)	80-mesh, 3 times (%)
0.420	97.4	5.4	4.4	0.4	—	98.4	2.7	1.2
.250	0.3	13.3	15.3	5.0	—	1.6	9.2	1.4
.177	0.2	13.1	15.0	10.1	0.1	—	12.2	—
.125	0.2	14.1	20.6	21.7	4.1	—	23.4	—
.074	0.4	19.6	20.4	27.1	37.0	—	24.9	6.4
Less than 0.074	1.5	34.5	24.3	35.7	58.8	—	27.6	91.0
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Mean, mm.†	—	0.132	0.150	0.112	0.063	—	0.130	0.043

* Material not ground.
† Particles retained on 0.420-mm. sieve neglected.

weight in terms of the dry weight. The atmosphere of 65 percent R.H. was that of a room, with humidity controlled within a tolerance of 2 percent R.H. and with temperature controlled at 21°C within a tolerance of 0.5°C. The atmospheres of 20, 79, and 90 percent R.H. were maintained by appropriate solutions in closed chambers located in the controlled-temperature room. These solutions were: for the 20 percent R.H. atmosphere, a 58 percent solution of sulfuric acid; for 79 percent R.H., a saturated solution of ammonium chloride; and for 90 percent R.H., a saturated solution of zinc sulfate [10].

The x-ray diffraction patterns were made with copper K_α radiation which was filtered by a 0.0012-in. nickel foil and made parallel by passage through a collimator containing a 0.020-in. pinhole. The specimen was attached by means of Scotch tape to the end of the collimator tube. For the unground sample the specimen consisted of a single clump of fibers. For the ground samples the powder was pressed into a Pyrex ring about 1-mm. thick and attached over the pinhole. The film distance was 6 cm. Before exposure the tube was operated for 30 minutes to assure a stable 16-milliampere current during exposure. All negatives were processed side-by-side in the same baths and with careful control of temperature. The completed patterns were photometered diametrically with a Leeds and Northrup recording microphotometer, great care being taken to assure comparability of the results from the different patterns. The photometric traces were reduced to a common base line and averaged over the two sides.

Fluidity, when measured, was determined according to the method described by Conrad and Tripp [2].

Results

The results of the particle-size analysis of the different samples are presented in Table I. While no significance is to be attached to the size distributions of the unground samples, the latter were subjected to the sieving operation and the results are given for completeness. Somewhat surprisingly, the material from the 2-mm. sieve seemed to have a larger proportion of fine particles than that from the 20-mesh sieve, which has a nominal opening of 0.84 mm. However, this is controverted by the hydrolysis results. This result, if it should represent the true picture, could have been influenced by differences in sharpness of the knives or speed of the rotors in the two mills used or by possible differences in moisture content at the time of cutting; on the other hand, an inaccurate analysis could have been caused by imperfections in the conditions of sieving referred to in the discussion of experimental procedure. It seems quite evident, however, that in the other cases the increasing severity of grinding resulted in progressively diminished particle size. This is shown especially by the mean size of particles computed for the material passing through the 0.420-mm. sieve—that is, for the major portion of each sample. Altogether, the results from sieving can be interpreted only in general terms.

Fluidity determinations were made on the series of ground samples derived from cut cotton and

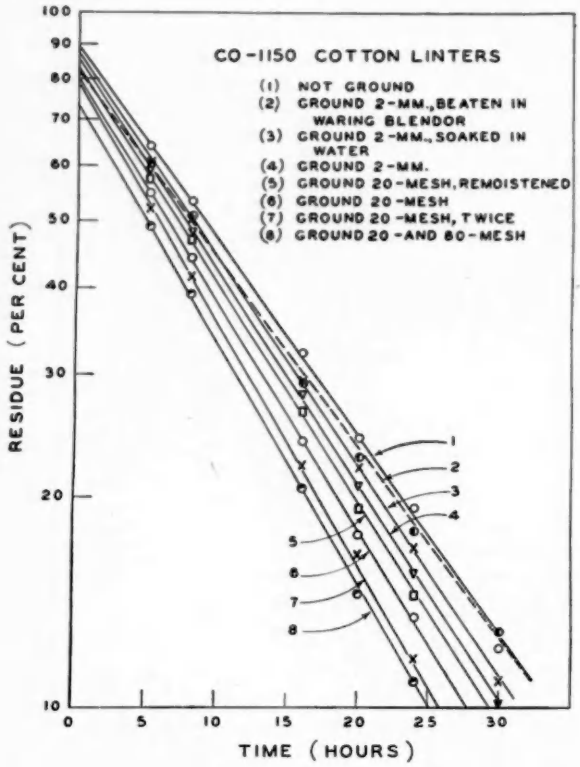

FIG. 1. *Curves showing rate of hydrolysis in 6N hydrochloric acid at 100°C of the crystalline fractions of variously treated portions of cotton linters.*

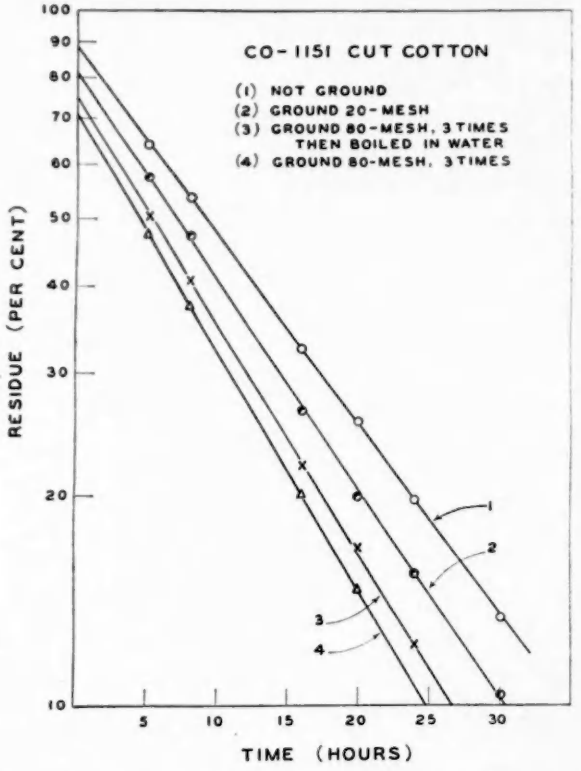
CO-1151 CUT COTTON
(1) NOT GROUND
(2) GROUND 20-MESH
(3) GROUND 80-MESH, 3 TIMES THEN BOILED IN WATER
(4) GROUND 80-MESH, 3 TIMES
RESIDUE (PER CENT)
TIME (HOURS)

FIG. 2. *Curves showing rate of hydrolysis in 6N hydrochloric acid at 100°C of the crystalline fractions of variously treated portions of cut cotton.*

showed no effect of the milder grinding (20-mesh sieve) as compared with the fluidity of the unground sample; but the more extreme conditions of grinding (grinding three times through the 80-mesh sieve) produced a small increase in fluidity, from 17.2 to 18.6 rhes.

Figures 1 and 2 show the rate curves for hydrolysis of the crystalline portion of the linters and cut cotton, respectively. No attempt was made to determine residue weights during the time interval when the amorphous phase was considered to be disappearing, as these data were not required for the crystallinity determination. In both figures it is evident not only that the intercepts of these curves on the zero-time ordinate are lower as the severity of grinding increases but also that in general the slope of the curves increases. A single exception is offered by the 2-mm. particles beaten in a Waring Blendor until fibrillation appeared, which obviously cannot be compared directly with the dry-ground samples. In addition to the increase in slope, the good linearity of the curves can be seen. The slope of the curve is, of course, a measure of the speed of hydrolysis and the steeper slopes for the more severely ground samples suggest that, by grinding, the mean crystallite size has been reduced, the available surface for hydrolysis has been increased, and therefore the rate must necessarily be increased. However, results of previous studies have indicated that wide variations of rate brought about by changes of temperature or acid concentration, but without any treatment that might change the crystallite size, do not cause a change in the zero-time intercepts. Therefore, a logical explanation of the decrease of intercept with increasing severity of grinding seems to be that the proportion of crystalline material is actually decreased, with a consequent gain in the proportion of amorphous material.

Table II gives the zero-time intercepts—that is, the degrees of crystallinity—for all samples of the two series, and accessibility for some of the samples of each series.

It will be noted that grinding, even through the largest (2-mm.) sieve, gave rise to an approximately 3-percent reduction in the estimated degree of crystallinity, and that as severity of grinding increased the

degree of crystallinity dropped correspondingly. For cotton linters the successive passage of the sample through the mill with 20- and 80-mesh sieves caused a reduction of 17 percent in the degree of crystallinity, whereas passage of the cut cotton three times through the mill with an 80-mesh sieve caused a 21-percent reduction in degree of crystallinity, based on that of the unground sample. Remoistening the 20-mesh ground linters restored the degree of crystallinity again by about 3 percent and remoistening the 2-mm. ground linters restored the crystallinity about 2 percent; heating the most severely ground cut cotton with water at the boiling temperature restored the degree of crystallinity by about 5 percent.

One possibility suggested by the downward shift of the hydrolysis rate curves by grinding was that as the particles were reduced in size some of them passed through the fritted glass filters, resulting in lower residue values. While it was not very probable that this would occur in such a way as to give the well-defined linear curves obtained, it was a possibility that was investigated. Accordingly, replicate 1-g. samples of cut cotton, ground through an 80-mesh sieve three times, were hydrolyzed for 1 hour and filtered through the fritted glass filter in the usual way. The filtrate was collected and passed through an ultrafine fritted glass filter (used for producing sterile filtrates by retaining bacteria on the filter). Any residue was washed with distilled water and dissolved in 72-percent sulfuric acid, and the cellulose was determined by the acid dichromate method [11]. The residues found in this way were 0.4, 0.2, and 0.0 mg., equivalent to 0.04, 0.02, and 0.00 percent of the original samples. These values are in great contrast to the 3- to 21-percent reduction of crystallinity through grinding (see Table II), and indicate that these losses cannot be ascribed to passage of the small particles through the filters.

TABLE II. DEGREE OF CRYSTALLINITY AND ACCESSIBILITY VALUES OF VARIOUSLY GROUND LINTERS AND COTTON

Material and degree of grinding	Degree of crystallinity	Degree of crystallinity lost (%)	Accessibility * (%)
Cotton linters			
Unground	0.88	0	5.8
Ground, 20-mesh	.80	9	7.6
Ground, 20-mesh, twice	.78	11	—
Ground, 20- and 80-mesh	.73	17	10.0
Ground, 20-mesh, remoistened	.83	6	—
Ground, 2-mm.	.85	3	—
Ground, 2-mm., soaked in water	.87	1	—
Ground, 2-mm., beaten with water in Waring Blendor	.82	7	—
Cut cotton			
Unground	.89	0	5.8
Ground, 20-mesh	.81	9	6.4
Ground, 80-mesh, 3 times	.70	21	8.4
Ground, 80-mesh, 3 times, boiled in water	.75	16	9.4

* Average of values by "method of slopes" and "method of heights" determined by Conrad-Scroggie modification [1] of Nickerson's hydrolysis-oxidation technique [13, 14].

The changes in accessibility values * given in Table II are mostly in accord with those for degree-of-crystallinity values, in showing a correlation with severity of grinding. As the fineness of the ground product increased, the percentage of accessible fraction increased—that is, the percentage of resistant (crystalline) cellulose decreased. The changes are smaller than those for the degree-of-crystallinity changes. Also, the cut cotton, which had been passed three times through the 80-mesh sieve, and then heated at 100°C with water, did not show the expected decrease in accessibility but actually showed a slight increase over that of the corresponding sample not heated with water. No accessibility measurements were made on the ground and remoistened cotton linters.

Since the accessibility values determined by the Conrad-Scroggie modification of the Nickerson technique are considered to be based on the oxidation of glucose itself and not on the oxidation of any filterable but incompletely hydrolyzed cellulose particles, this is further evidence from a complementary direction that grinding does increase the amorphous fraction at the expense of the crystalline fraction.

In Table III are given the integral heats of wetting and the moisture regains for most of the series of samples listed in Table II. The degree-of-crystallinity figures of Table II are repeated for comparison.

It will be seen that the integral heat of wetting in-

* It is recognized that "accessibility" measurements involve not only the amorphous fraction but also the surfaces of the crystallites—that is, all regions of the cellulose fiber that are accessible to the hydrolyzing agent. One would therefore expect the accessibility to be greater than the amorphous fraction by an amount that is related to the total surface area of the crystallites. In the present instance, the accessibility as measured by the Nickerson technique is smaller than the amorphous fraction derived (by difference) from the acid-hydrolysis crystallinity measurements. An explanation may lie in differences in the technique for determining the zero-time intercepts by the two methods.

TABLE III. VALUES OF INTEGRAL HEAT OF WETTING AND MOISTURE REGAIN FOR UNGROUND, GROUND, AND GROUND AND RE-WET SAMPLES

Material and degree of grinding	Degree of crystallinity	Integral heat of wetting (cal./g.)	Moisture regain at 20% R.H. (%)	65% R.H. (%)	79% R.H. (%)	90% R.H. (%)
Cotton linters						
Unground	0.88	7.74	2.80	6.18	8.96	10.64
Ground, 2-mm.	.85	9.69	2.86	6.25	8.70	10.60
Ground, 20-mesh	.80	10.90	2.84	6.52	8.96	10.91
Ground, 20-mesh, twice	.78	—	3.10	6.67	9.30	11.00
Ground, 20- and 80-mesh	.73	11.76	3.08	6.99	9.49	11.48
Cut cotton						
Unground	.89	8.37	2.79	5.76	8.98	10.48
Ground, 20-mesh	.81	10.14	3.02	6.17	9.10	10.74
Ground, 80-mesh, 3 times	.70	11.94	3.39	7.32	10.14	11.86
Ground, 80-mesh, 3 times, boiled in water	.75	9.91	3.38	6.99	10.26	12.24

creased with severity of grinding in all cases. For the cotton linters the integral heat of wetting increased from 7.74 to 11.76 calories per gram, or by about 52 percent, as a result of grinding successively through the 20- and 80-mesh sieves. The integral heat of wetting for the cut cotton increased from 8.37 to 11.94 calories per gram, or about 43 percent, as a result of grinding three times through the 80-mesh sieve. The integral heat of wetting of the latter sample dropped to 9.91 calories per gram as a result of heating with water and drying; this is still about 18 percent above the value for the unground sample. The sample of cut cotton which was passed three times through the 80-mesh sieve also showed the largest heat of wetting, although this was not much above that for the cotton linters ground successively through the 20- and 80-mesh sieves. This is in good agreement with the changes in degree of crystallinity and also agrees with the difference in relative particle size indicated in Table I for these two samples.

The integral heats of wetting of the different samples were plotted against the degree of crystallinity of the same samples, with the results shown in Figure 3. While the relationship is by no means perfect, it is evident that a general inverse relationship exists. It also seems probable that this relationship is not linear. If the curve is extrapolated to intersect the crystallinity axis it is evident that the heat of wetting would be near zero at complete crystallinity, as might be predicted from other considerations.

The moisture-regain values listed in the last four columns of Table III show a fairly consistent over-all increase as the severity of grinding increases, with some minor exceptions. Thus, the relative increases in moisture regain of cotton linters ranged from 6 percent at 79 percent R.H. (8.96 percent to 9.49 percent) to 13 percent at 65 percent R.H. (6.18 percent to 6.99 percent) on going from the unground material to the most severely ground. The relative gains were somewhat greater in the case of the cut cotton, ranging from 13 percent at 79 percent and 90 percent R. H. (8.98 percent to 10.14 percent, 10.48 percent to 11.86 percent) to 27 percent at 65 percent R.H. (5.76 percent to 7.32 percent). The severely ground cut cotton that was boiled in water showed the expected decrease in moisture regain at 65 percent R.H., but at 79 percent and 90 percent R.H. exceptions were noted.

The moisure regains at 20, 65, 79, and 90 percent

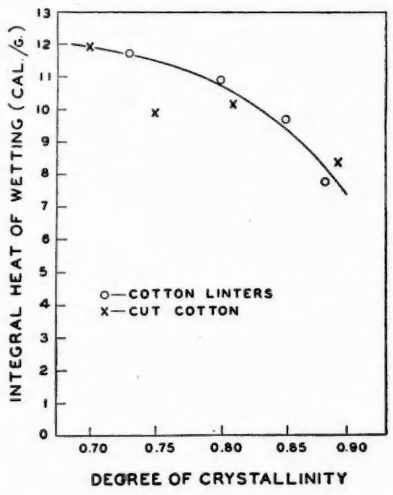

FIG. 3. *Relationship between degree of crystallinity and heat of wetting of ground samples.*

R.H. were plotted against degree of crystallinity of the same samples, with the results shown in Figure 4. It will be noted that a fair degree of correlation exists under all four atmospheric conditions. At the lower relative humidities (20 percent and 65 percent R.H.) the plotted values exhibit good linearity; the values are somewhat scattered at the higher relative humidities but appear to be linearly related.

X-ray diagrams were made only of the cut cotton. The averaged curves derived from the photometric traces of the diffraction patterns are reproduced in Figure 5. They do not show evidence of any noticeable change in the crystalline structure. There is no evidence of the development of an amorphous halo in pattern C, as might be expected on the basis of the hydrolysis value for this sample as compared to that corresponding to pattern A. Neither is there evidence of the formation of any hydrate cellulose in pattern D, as might have been expected on the basis of studies by Hess and coworkers [7] and of Hermans and Weidinger [6]. Of course the relative magnitudes of the indicated crystallinity changes are comparatively small in the present case and it is well known that x-ray patterns are not suitable for making small distinctions of a quantitative nature.

Some other data may be introduced at this point for the bearing they have upon the interpretation of the results. Samples of the cotton linters were hydrolyzed in the usual way for crystallinity determinations except that 2.5*N* hydrochloric acid was employed (instead of 6*N*) in order to reduce the rate

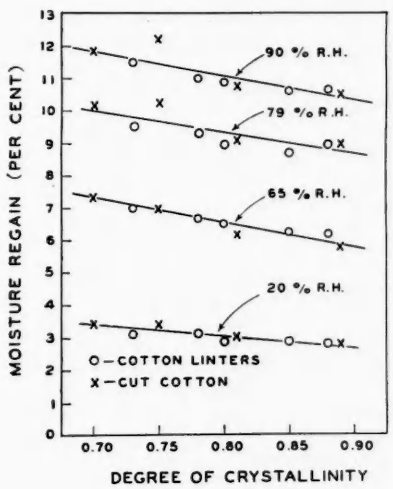

FIG. 4. *Relationship between degree of crystallinity and moisture regain of ground samples.*

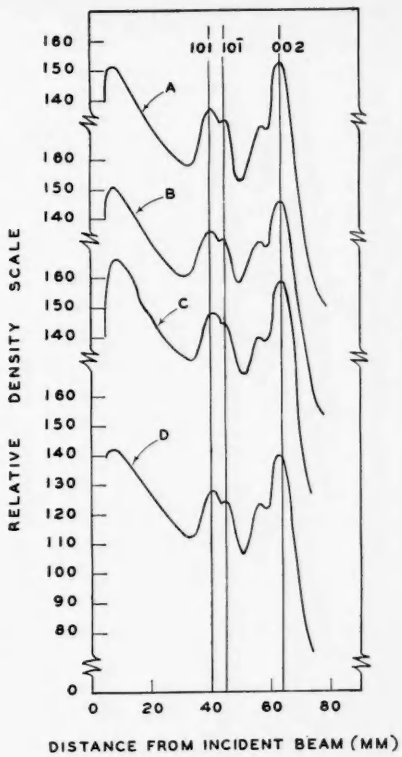

FIG. 5. *Relative density along the equators of the x-ray diffraction patterns of samples of cut cotton. A—Not ground. B—Ground through 20-mesh sieve. C—Ground three times through an 80-mesh sieve. D—Same as C but heated with water at 100°C and dried.*

of hydrolysis. The hydrolysis was interrupted, and the residues were separated from the hydrolysis mixtures and washed according to a predetermined time schedule. The residues were dried in a vacuum oven and their weights determined. They were then used for fluidity measurements. The residue weights, expressed as percentages of the starting weights, the fluidities, and the estimated degrees of polymerization, are shown in Table IV.

From Table IV it will be seen that after only 15 minutes of hydrolysis the fluidity of the material had changed from 9.90 to 54, and the estimated degree of polymerization from 1,225 to 195. At this stage, however, only 2 percent of the substance had been lost through hydrolysis. This 2-percent loss must have come almost entirely from the 12 percent of amorphous material, since under the conditions of acid concentration and temperature used the rate constant (based on other experiments) would allow for less than 0.2 percent crystalline cellulose hydrolyzed. The change in fluidity during the re-

TABLE IV. PERCENT OF WEIGHT REMAINING, FLUIDITY AND ESTIMATED DEGREE OF POLYMERIZATION OF RESIDUES OF COTTON LINTERS AFTER DIFFERENT PERIODS OF HYDROLYSIS WITH 2.5*N* HYDROCHLORIC ACID

Time of hydrolysis with 2.5*N* HCl at 100°C	Weight of original sample remaining	Fluidity of residues $c = 0.5$ g./100 ml. $G = 500$ sec.$^{-1}$	Estimated degree of polymerization (glucose units)
(hrs.)	(%)	(rhes)	
0.0	100	9.90	1,225
0.25	98	54.0	195
0.50	96.6	54.4	191
1.0	95.1	54.1	194
3.0	92.7	55.0	185
7.0	88.4	54.7	188

mainder of the 7 hours is practically negligible, in line with the finding of Davidson [3]. During this latter interval it is computed that about 70 percent of the amorphous and 4 percent of the crystalline cellulose has disappeared. From the rapid lowering of the degree of polymerization in the initial few minutes it must be inferred that the hot acid penetrates the fiber material exceedingly rapidly. This would appear to be in the amorphous regions, especially as the succeeding 6¾ hours causes scarcely any additional change in degree of polymerization. It may be interpreted that the rapid hydrolysis breaks the chains between the crystalline areas, releasing the crystalline particles, which then hydrolyze by progressive surface attack. Furthermore, it is suggested by the fact that the degree of polymerization does not decrease appreciably further after the first few minutes that hydrolysis does not take place appreciably from the ends of the crystallites but, rather, largely from the lateral faces.

Discussion

While the results presented cannot be taken as proving conclusively the proposition of a reduced degree of crystallinity as a result of grinding, it is believed that they are, in general, consistent with this point of view. The hydrolysis rate curves for those portions of the samples which are interpreted to represent the crystalline fraction display a high degree of linearity and obey the laws of a first-order reaction. When extrapolated to zero time the intercepts, which are interpreted to represent the crystalline portion at the beginning, occur at successively lower percentages as the severity of grinding increases. That this lowering of the curves is not due to a loss of residue by passage of the smaller particles through the filter is proved both by the negative results obtained on the filtrate by the ultrafine filter and by the results with the Conrad-Scroggie modification of the Nickerson technique, which dealt not with the residues but with the glucose formed during hydrolysis.

The increasing slope of the hydrolysis rate curves representing the increasing rate of hydrolysis as severity of grinding increases is consistent with the view that the crystalline areas are reduced in size by the grinding process, giving rise to a greater total surface. The rate of hydrolysis of the crystalline portion would be expected to be proportional to the surface exposed to the hydrolytic agent. On the other hand, it has been shown in previous studies that the rate can be changed over a wide range by changes in concentration of acid and in temperature of the hydrolytic mixture without any change occurring in the intercept at zero time. Thus, when acid concentration and temperature are kept constant, a change in rate of hydrolysis of the crytalline fraction is not, in itself, an indication of difference in proportion of crystalline fraction but rather of the size of the particles. On the other hand, a change in the position of the intercept should be interpreted as a change in the proportion of the crystalline and amorphous fractions.

With only a few exceptions the remainder of the auxiliary data supports the interpretation placed on the hydrolysis data. The integral heats of wetting, which are presumed to be dependent on the amount of surface exposed to moisture, are seen to increase with increasing severity of grinding. Since it is generally agreed that water cannot penetrate within the crystalline lattice of native cellulose, this must mean an increase in accessible surface. Likewise, the reduction of the integral heat of wetting after the most severely ground sample had been remoistened is consistent with the idea of a reduction of accessible surface, presumably by recrystallization. The moisture regains increase, too, corresponding to the increased severity of grinding, and show a like recession after rewetting and exposure at 65 percent R.H., though not at 79 percent or 90 percent R.H.

The x-ray diffraction patterns have not confirmed the interpretation of reduced crystallinity as a result of grinding, which is indicated by the other data.

It is not certain how much importance should be placed on these findings in view of the fact that it is the crystalline phase that is principally responsible for the pattern and the crystalline phase is still indicated to be greatly in excess of the amorphous in the most severely ground samples. The grinding in the present studies was much less severe than that employed by Hess and coworkers [7] and by Hermans and Weidinger [6], in which complete destruction of the crystalline x-ray pattern was observed. Also, the small change of fluidity brought about by the most severe condition of grinding employed is evidence for the relatively mild grinding, as compared with the results of Hess and coworkers, in which the viscosity after 2 hours had decreased to one-half the original value.

It seems difficult to explain the fluidity changes obtained with the hydrolysis residues from cotton linters, where after only 15 minutes of hydrolysis the fluidity had increased almost to its maximum value, except on the basis that an amorphous network connecting and surrounding the crystalline areas is ruptured with great speed, whereby the crystalline particles, perhaps with a fringe of broken extended chain ends, are liberated into the hydrolytic medium. At this stage, and under the conditions employed, only 2 percent of the residue had disappeared. However, the fluidity values indicate a mean chain length of approximately 195 glucose units. If the picture of the cellulose structure frequently presented is true, whereby single long cellulose chains pass successively through a number of crystalline and amorphous areas, the crystalline regions must protect the internally reposed chains but allow the connecting amorphous network to be attacked at once. The implication would be that the crystalline areas have a mean length of roughly 195 glucose units, although it is admittedly possible for some recrystallization of fringe regions to occur even during rapid hydrolysis. The fact that the degree of polymerization does not change appreciably during the remainder of the 7-hour hydrolysis period suggests that in subsequent hydrolysis the crystallites liberated from the network structure during the initial period are attacked largely from the sides and only slightly from the ends. On the other hand, the reduction in crystallinity, indicated by the hydrolysis results on the variously ground material, suggests a fragmentation of the crystalline areas by the grinding action. This may be pictured as occurring by communication of the grinding forces to the amorphous chain network, which in turn exerts forces from various directions to the ends of the crystalline regions. These ends, being the points of greatest susceptibility, are opened up more or less zipperlike to give rise to new surfaces, as well as newly separated cellulose chains which have become dislodged from their crystal lattices and which, owing to the still clinging amorphous network, are not able to reparticipate in the crystal structure. It is entirely possible that crystal structure could be reduced through this mechanism out of all proportion to the visible reduction of particle size, as observed under the microscope. This could readily account for the increases of amorphous material, indicated by the present hydrolysis results as well as by observations of other workers in this most interesting field.

Summary

In order to study the effect, previously noted, of dry grinding in Wiley mills on the degree of crystallinity of the cellulose as determined by the hydrolysis-residue method, samples of cotton linters and cut cotton which had been previously purified were reduced to small particles under conditions of increasingly severe grinding.

An analysis of the particle size of the resulting material in a standard set of sieves, which, owing to the nature of the material, was only partially successful, indicated that the particles became definitely smaller as the severity of the grinding increased.

It was found that even mild grinding—for instance, passage through a Wiley mill equipped with a sieve having 2-mm. openings—affected the indicated degree of crystallinity to an appreciable extent and that as the severity of grinding became greater the degree of crystallinity dropped correspondingly. This was accompanied by only a slight change in degree of polymerization in cases of the most severe grinding. It was also established that wetting the samples and allowing them to dry resulted in a partial regain of the indicated degree of crystallinity lost by grinding.

By means of a bacterial filter, it was shown that the reduction in indicated degree of crystallinity with reduction in particle size was not due to increasing amounts of the finer particles passing through the filters ordinarily used. Furthermore, a progressively increasing slope of the linear log residue-time curves with increasing severity of grinding corresponds to

progressively greater rates of hydrolysis and suggests a decreasing crystalline particle size.

The results of hydrolysis rate measurements were correlated with accessibility measurements (based on liberated glucose), heat-of-wetting measurements, moisture adsorption determinations, x-ray diffraction patterns, and fluidity measurements made on the same samples. Except for the x-ray diffraction patterns, these largely supported the interpretations of the hydrolysis-residue results—namely, that grinding causes an increase in the amorphous fraction at the expense of the crystalline fraction. Although the x-ray patterns were not changed noticeably by the grinding, it is assumed that the extent of the change was insufficient to be reflected in the patterns.

The results of the present study are interpreted in terms of a continuous network of more or less amorphous cellulose chains connecting crystalline cellulose areas. The observation that only 15 minutes after the beginning of hydrolysis in 2.5*N* hydrochloric acid cotton linters attain almost their maximum fluidity, which scarcely changes for the following 6¾ hours, whereas in 15 minutes only 2 percent of the residue has been hydrolyzed away, is evidence that the crystalline particles are liberated very quickly, through breaking and partial hydrolysis of the amorphous network, and that hydrolysis thereafter takes place principally by surface attack and largely from the lateral surfaces. The estimated degree of polymerization of about 195 glucose units after this short period suggests that this is probably the order of magnitude of the crystallite length. On the other hand, it is postulated that, by grinding, forces are communicated through the amorphous chains to the ends of the crystalline areas, causing splitting and the development of new loose amorphous chain structures.

Acknowledgment

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Compressional Behavior of Textile Materials

I. Measurement at a Constant Rate of Deformation*

Rogers B. Finch†

Abstract

The description of an instrument to measure the compressional characteristics of textile materials at a constant rate of compression and recovery, utilizing the high speed and sensitive response characteristics of a resistance-wire strain gage cantilever load cell, is given. It employs a high-speed, sensitive, rectilinear, electronic recorder in conjunction with the strain gages in an amplified and rectified A.C. Wheatstone bridge. Reversal of the direction of travel of the deforming foot is obtained by use of manual- or limit-switch controlled magnetic clutches. A wide range of load scales and traveling foot speeds may be obtained. Response curves are given for a variety of textile materials to indicate their behavior characteristics and illustrate the many ways in which the instrument may be employed.

A. General Aspects of Compressional Behavior

1. Historical Interest in Compressional Behavior

The textile industry has long been interested in the compressional behavior of bulk fiber and of fabrics [39]. Studies of this type of behavior have usually been for such purposes as comparison of the ability of various types and blends of fibers to perform satisfactorily as fillers for sleeping bags, pillows, mattresses, comforters, insulation, etc. [7–16]; evaluation of the "thickness" of fabrics [1–6]; comparative evaluation of floor coverings and pile fabrics for other uses with respect to their ability to resist deformation processes [17–25, 33]; and determination of the ability of fabrics to resist creasing, crushing, etc. [26–30]. In most cases, however, the criteria developed to describe such behavior have been dependent upon the test method employed and the definitions of the quantities measured. For example, there has arisen the use of the term "resilience," some of the definitions of which have recently been reviewed by J. H. Dillon [31] and which has been employed by H. Mark [12] in his recent approach to the relationship between the subjective property descriptions used in the industry and the properties of the fibers themselves.

The use of the term "resilience" results in a number of difficulties inasmuch as its origin, stemming from the industry itself, is not quantitative and the attempts to give it quantitative definition have not been entirely satisfactory. Harold DeWitt Smith [41] has suggested a definition of this quantity based upon the similar term employed for metal behavior. Unfortunately, the behavior of textile materials is quite different and necessitates a completely different approach to the entire field of the mechanical behavior of this class of materials.

2. Fabric Structure and Behavior

In its most complex, but most common, form a textile material is a fabric composed of yarns in a particular weave. The yarns are, in turn, composed of fibers of a given statistical distribution of dimension and space relationships. The fibers, likewise, are composed of high polymeric molecules in a quasi-crystalline or micellar network which, in the case of some natural fibers, form larger associations of submicroscopic dimensions within the fibers known as fibrils. Thus the fabric may be characterized as being composed of long-chain molecules, whose mechanical and thermodynamic inter-relationships are governed by definite and predictable laws [12, 32, 35, 37], placed in a geometrical space distribution governed by the laws of botanical or synthetic origin and the mechanical laws of yarn and fabric manufacture. It can be seen from this qualitative analysis that the performance of the fabric, by whatever means it is evaluated, is a reflection of these inherent properties and the geometrical "form factors" by which these

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Part II of this series, entitled "Measurement of Compressional Stress Relaxation at Constant Deformation," will appear in the April issue.

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properties are modified. Such modification can be so great as to totally obscure the nature of the fiber constituting the material.

Attempts have recently been made to relate the ultimate performance of a fibrous aggregate to the deformation processes of molecular aggregates [32]. This approach assumes that various deformation processes are related to the viscoelastic nature of the molecular aggregates, and behavior of materials has been described in terms of the constants associated with the elements of a more or less complex mechanical viscoelastic model. It is true that materials may be represented in such terms and that the comparison of the constants thus obtained may be of considerable value in the industry when it is possible to translate these values into terms associated quantitatively with the subjective terms now used. The ability to obtain such a translation is entirely dependent upon the ability both to measure the constants associated with the viscoelastic model of the material and to interpret the descriptions of the properties of materials used in the industry by skilled textile experts upon whose judgment depends the acceptance or rejection of practically all industrial materials. The key to this problem is the ability to measure behavior, and it is one aspect of this problem to which this series of papers is devoted.

The textile material designer or engineer has, however, an additional problem. It is his concern not only to provide a material acceptable to the consumer in terms of his subjective interpretation but also to engineer the material efficiently in terms of the fibers, yarn structure, and fabric structure which will best meet these requirements. He must, therefore, be able to select the best combination of fiber, yarn structure, and fabric structure which will result in the viscoelastic behavior desired, if only the mechanical properties are considered. This means that he must have available as engineering data a tabulation of the viscoelastic properties of the fibers and the laws of modification of these properties which takes place in a fabric ("form factor" modification). Few attempts have been made to develop such "form factor" laws but those made by Womersley [43] and those suggested by F. T. Peirce [38] are directed toward the description of the differential geometric relationships existing between deformed fabrics and their component yarns. This approach must be expanded to include the laws of molecular-aggregate kinetics as well as the differential geometry of the micro- and macro-structures represented by the fabric and its components if the ultimate aim of research—that is, the prediction of the engineering properties of materials based upon sound physical and mathematical laws—is to be achieved.

3. Measurement of Behavior

Thus far, there has been little attempt, at least as evidenced by the literature, to approach textile-material behavior from this over-all viewpoint. Ordinarily, the approach has consisted of investigating a particular material by a particular method for a specific end purpose, and the steps employed have been the selection of a variety of materials to be evaluated, the selection of an appropriate instrument for measurement (or development of one if none in existence were appropriate), and the comparison of the materials based on the measurements obtained from the particular instrument. It may be said that this type of investigation is satisfactory for a particular purpose and, undoubtedly, in the absence of a better basis, much more investigational work will be done along these lines. This is the empirical approach and the end result is that no significant contribution can be made to the general fund of knowledge from which the character of general laws of behavior might be drawn, because the published literature ordinarily fails to present clearly the details of measurement, the statistical basis on which the experiments were designed and the details of the analysis of the materials investigated. The lack of details of measurement and of the statistical basis is the fault of the investigator, but the lack of details of the analysis of the materials is due to deficiency in research instruments, in techniques for measurement, and in interpretation of results.

This situation gives rise to a vicious circle; the keynote to the resolution of this circle is an appreciation of the fact that laws common to all such behavior do exist and that adequate instrumentation for investigating them through the medium of materials carefully selected for that purpose must be developed wherever research or developmental work of any nature is undertaken. Only through a coordination of effort can the empirical approach be replaced by the universal.

4. Interpretation of Behavior

The laws of molecular-aggregate kinetics have been approached both from the standpoint of the behavior

of individual molecules subjected to certain thermodynamic and deformation conditions [12, 36, 37] and from the standpoint of the behavior of individual fibers or filaments in creep or in stress relaxation [35]. These behaviors are related through the geometry of the fiber composed of molecular aggregates although little has as yet been done to clarify this relationship. In fact, adequate instruments for such measurements were not available prior to those developed in the Slater Memorial Research Laboratory of the Textile Division of the Massachusetts Institute of Technology [40]. The results of the observations made on materials subjected to various fundamental deformation processes by the use of such instruments indicate that these processes in textile materials are governed by definite laws which relate the loads applied, the resultant deformations, the time elapsed, the rate of deformation, and the deformation history of the material. Observations of the behavior of a material are, then, directly related to the conditions imposed upon it by the instrument. These considerations led to the analysis of behavior from creep and stress relaxation studies wherein the conditions of load or of deformation remain constant while the material itself responds with time in accordance with its previous deformation history and its viscoelastic nature. Hence constant rate of deformation and constant rate of load, or similar types of tests, are simply methods of deforming the material so as to cause it to perform in accordance with the conditions imposed. These tests are generally of some value because they provide reproducible methods for the comparison of the empirical behavior of materials, although the results are difficult to analyze in terms of the fundamental rate processes since they represent the superimposition in time of differential increments of stress relaxation, imposed on the material in a relatively arbitrary, but complex, manner. However, the literature contains many papers based upon investigations of this type, and it is not surprising, therefore, to find the variety of concepts which have arisen—for example, in connection with compression testing. It can be seen, then, that the definitions of such terms as "resilience" as indicated by J. H. Dillon [31], and "thickness" and "crease-resistance" as indicated by H. F. Schiefer [5, 29, 30] are the result of particular methods of deforming the material and observing its response.

The laws of the geometry of deformation processes applied to a complex structure such as a fabric are yet to be elucidated. The behavior of a material is exceedingly complex when examined in terms of its components and their properties. F. T. Peirce [38] has suggested that certain simplifying assumptions in connection with the derivation of such laws should make possible a reasonably simple approach where, otherwise, unreasonably complex mathematics might result.

B. Instruments for Measuring Compressional Behavior

1. Theory of Instrument Design

It has been pointed out that in order to investigate these laws of inherent behavior and of geometrical form it is necessary to develop instruments to measure performance which do not impose their own characteristics upon the observed behavior of the material in a manner which will prevent the use of the results for the development of these laws. The basic requirements of any research instrument are twofold: the errors inherent in its operation must be fewer than those of the material being measured, and the design must be such that errors due to personnel operation are reduced below those of the material being measured. The combination of these requirements necessitates the collaboration of the instrumentation engineer and the textile technologist.

After a series of investigations in the Massachusetts Institute of Technology Textile Division Laboratories [20, 28], it was determined that rates of response to deformation of textile materials were much higher than the techniques which were then available could measure. Where mechanical balances were used, reversals of deformation direction caused a lag in the instruments due to their own mechanical inertia. It was determined that a load-recording system was desirable which had a high rate of response but no appreciable deformation under load. These requirements were admirably met by the use of resistance-wire strain gages used in conjunction with a cantilever-type weighbar described by Hindman and Krook [34]. The Division was then faced with the question of selecting an appropriate method of application of deformations to textile materials.

2. Deformation Rate Processes of the Instrument

To meet research and educational requirements, it was felt that such an instrument should be com-

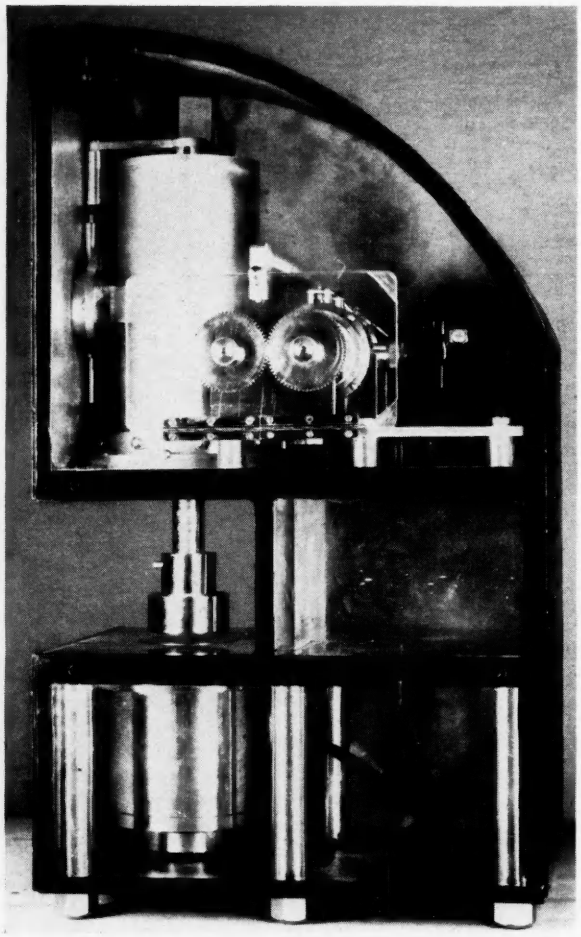

FIG. 1A. *Picture of the compression unit showing the locations of the load cell, the traveling foot, the anti-backlash gears, the change gears, the magnetic clutches, and the driving motor.*

paratively flexible with respect to rates of deformation as well as to the absolute deformations available in order to accommodate a wide variety of textile materials. This was done so that the instrument could be applied to empirical but reproducible comparisons of the performance of fabrics of the same general commercial category and could also serve as a means of exploring the fundamental deformation processes of materials whose structures are related. The actual rates chosen are given in the description of the instrument which follows, but it should be pointed out that the range of rates chosen was dictated by the response rate of the particular recording system utilized. The consideration of the size and shape of the deforming foot was then undertaken. After considerable investigation of the effects of different sizes and shapes, a circular foot, 2¼ inches in diameter, was selected since this particular size and shape minimized the edge effects which tend to occur whenever a restricted portion of a test specimen is deformed. The circular shape produced the smallest perimeter for a given foot area and the value of the area chosen was such that a large area of the specimen could be considered to enter into the deformation.

C. Description of the Instrument

The instrument itself is composed of two parts: a compression mechanism and a control-recording unit.

1. The Compression Mechanism

The compression unit is used for the purpose of deforming the specimen in some desired manner and recording the force transmitted through the specimen as time progresses. The principle of design of the compression unit is indicated in Figure 1A. It consists, first of all, of an upper head which is a rigid part of the frame of the instrument. This upper head contains rigid guides within which the deforming foot operates. The motion of the foot is

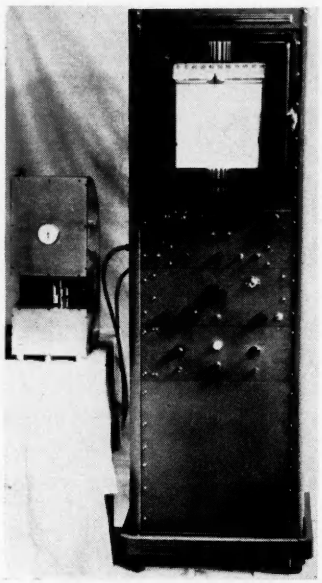

FIG. 1B. *Photograph of the complete compression apparatus showing the compression unit and the control-recording unit. The recorder is at the top of the panel. The panel below it controls the manual or automatic cycling and the overload reset. The middle panel contains the bridge-balancing controls. The lower panel contains the main power controls. The large bottom panel is provided for the addition of the integrator.*

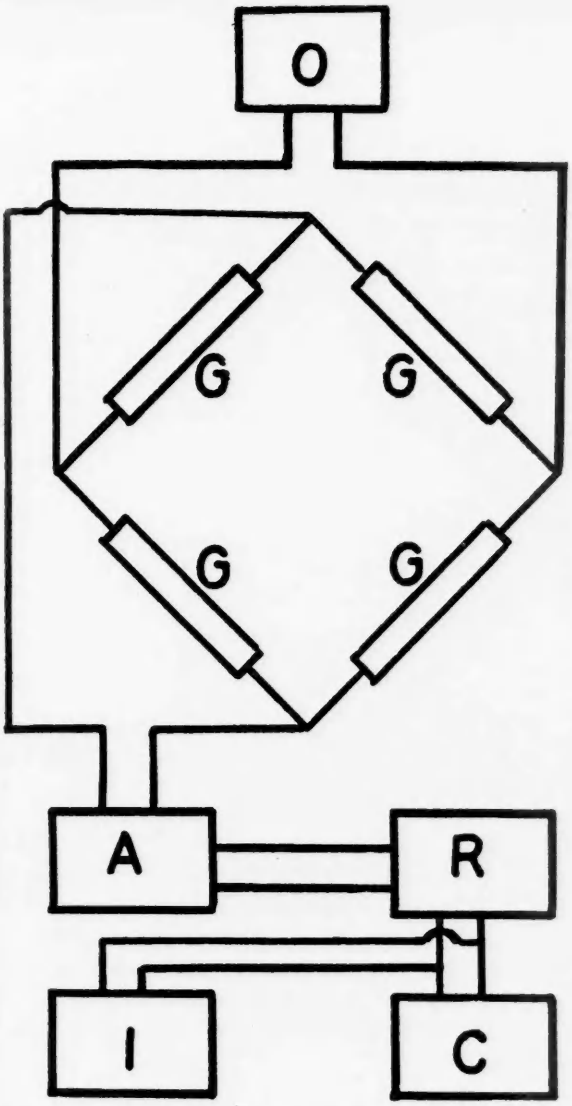

Fig. 2. *Schematic diagram of the recording and analyzing systems. G—strain gages, O—audio-oscillator, A—amplifier, R—rectifier, C—chart recorder, and I—integrator.*

controlled by a synchronous electric motor driving through a pair of reversing magnetic clutches and a system of gears which have been precisely machined to very close tolerances to hold errors due to backlash to a value which is less than the smallest readable division of the dial gage. The dial gage gives the position of the bottom face of the traveling foot relative to the top face of the lower head on which the specimen rests. Thus the amount of penetration into the specimen may be determined to the nearest thousandth of an inch. The rate at which the circular foot travels toward or away from the upper face of the lower head is controlled by change gears, which may be varied for a wide range of rates (0.01, 0.02, 0.05, 0.10, 0.20, 0.50, and 1.00 inch per minute) between the magnetic clutches and the helical antibacklash gear. Control of direction of travel is produced through the magnetic clutches, one of which is engaged as the other is released, as determined by the recording-control unit.

The force transmitted by the specimen causes the lower head to deflect less than a thousandth of an inch, thus causing a change in separation of the upper and lower heads which is less than the smallest division of the dial gage. Such a small deflection does not significantly affect the rate of deformation as determined by the change gears. This lower head is rigidly connected to a steel cantilever-type load cell [34] to which are cemented resistance-wire strain gages. Although the motion of the lower head, and thus the end of the cantilever load cell, is extremely small, it is sufficient to cause measurable changes in the resistances of the strain gages. This change in resistance is linear with the load applied to the lower head in accordance with the design of the cantilever and the placement of the gages. The calibration may be checked at any time by pulling out the knob at the bottom of the front panel and placing a 1-pound weight on the load cell. This produces a change in resistance equivalent to a 1-pound load, permitting the control-recording unit to be calibrated accordingly.

2. The Control-Recording Unit

This unit is used for the complete operation of the assembled apparatus. It consists basically of two parts: switches for controlling the reversing magnetic clutches, and the recorder (see Figure 1B).

The clutches may be reversed in three different ways. There are, first of all, manual switches for reversing the clutches in any manner desired by the operator. There are limit switches which cause the clutches to be reversed at any desired value of load, determined by the position of the limiting pointers on the scale at the top of the recorder. If cycling of the specimen between two load limits is desired, these eliminate the factor of operator error and free the operator for other duties while a long-time test is in progress. A counter is provided which records the number of completed cycles so that the operator may determine which cycle is in progress without unrolling the recording chart to count them all.

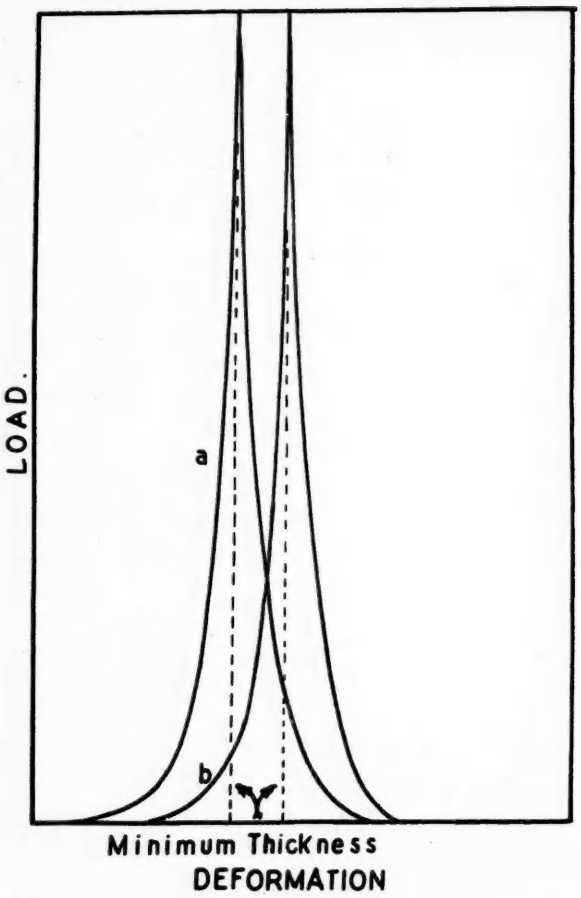

FIG. 3. *Comparison of the shape of load-compression curves for the first (a) and conditioned (b) cycles obtained from a blanket fabric. The first portion of each curve indicates the compression portion of the cycle. The dashed line is the minimum thickness to which the fabric was compressed. The second portion of each curve indicates the compression recovery.*

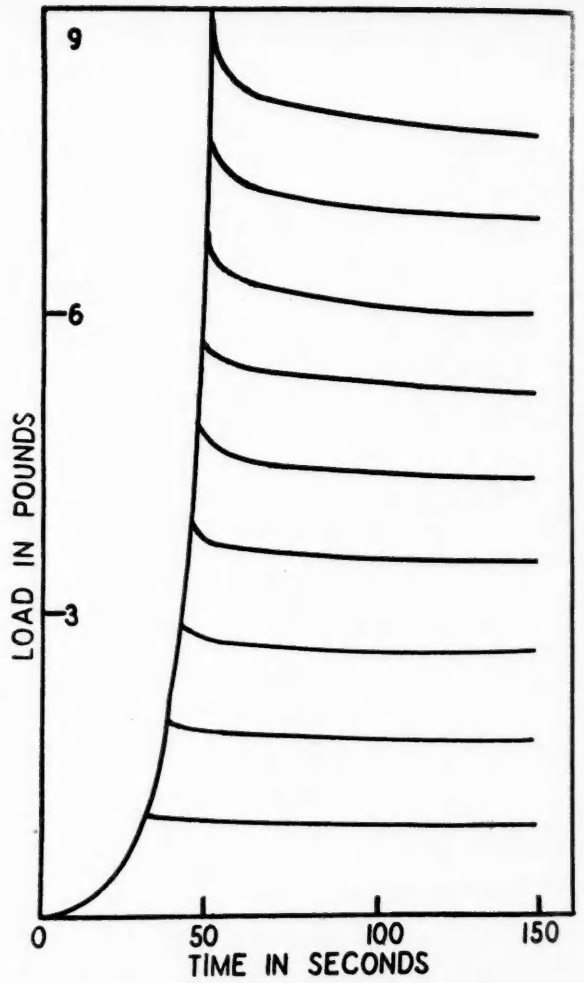

FIG. 4. *Comparison of load-relaxation curves at different compression loads for the conditioned cycle of a blanket fabric. The first portion of the curve represents the compression part of the conditioned cycle. The relaxation curves which follow the compression curve were obtained at nine different places in the fabric in order to minimize load-history effects.*

An overload safeguard is also provided on the recorder so that the scale cannot be overrun because of the failure of the operator to reverse the mechanism in time. If the end of the recorder scale is reached, a limit switch is tripped which causes the entire unit to stop. A further safeguard is provided on the zero end of the scale. The traveling foot can travel upward only until it trips a limit switch, thus causing the entire unit to stop. It cannot be started again without pressing a restart switch and throwing the reversing switch.

The strain-gage-recorder-integrator circuit is indicated schematically in Figure 2. The gages form the legs of a Wheatstone bridge which is excited by an alternating current source having a frequency around 250 cps. This bridge may be balanced at any time by adjusting the tuning eye on the front panel to a minimum position. The change in resistance of the gages due to application of load thus produces a voltage change across the output of the bridge. This voltage is fed into an A.C. amplifier, rectified, and fed into the recorder. A selector switch is provided which allows use of the full scale of the recorder for 1, 2, 5, 10, 20, or 50 pounds as desired. The scales are of essentially equal precision as the cantilever is designed to be linear over this entire range with a considerable factor of safety.

The recorder is of the continuously recording potentiometer type. Several such instruments are commercially available and the one employed in this tester is the Leeds and Northrup, type G. It em-

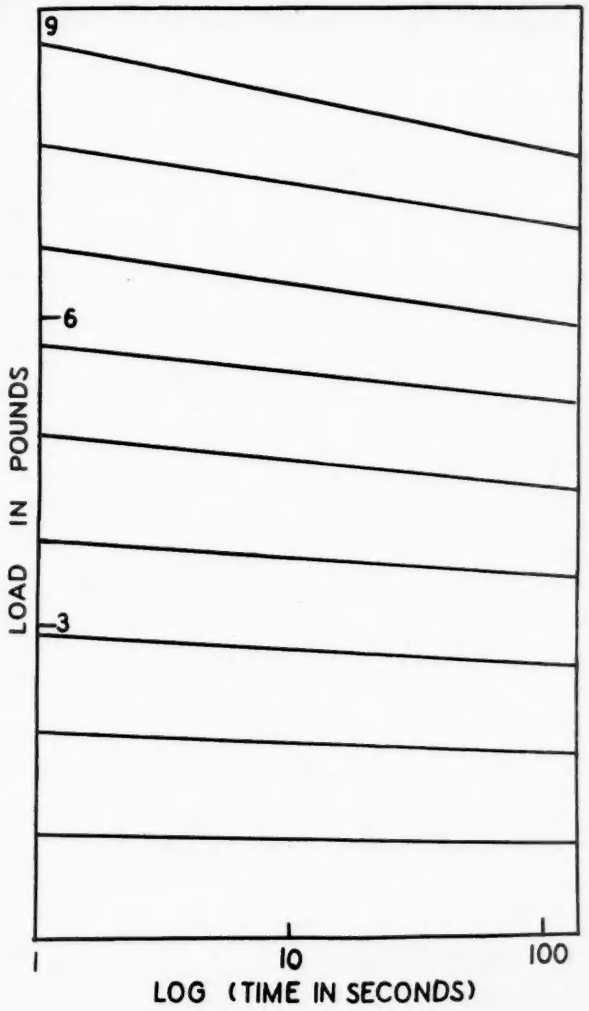

Fig. 5. *Comparison of load-relaxation curves obtained in Fig. 4 where the time scale has been converted to a logarithmic one. On such a diagram the relaxation curves appear to be linear in the range investigated.*

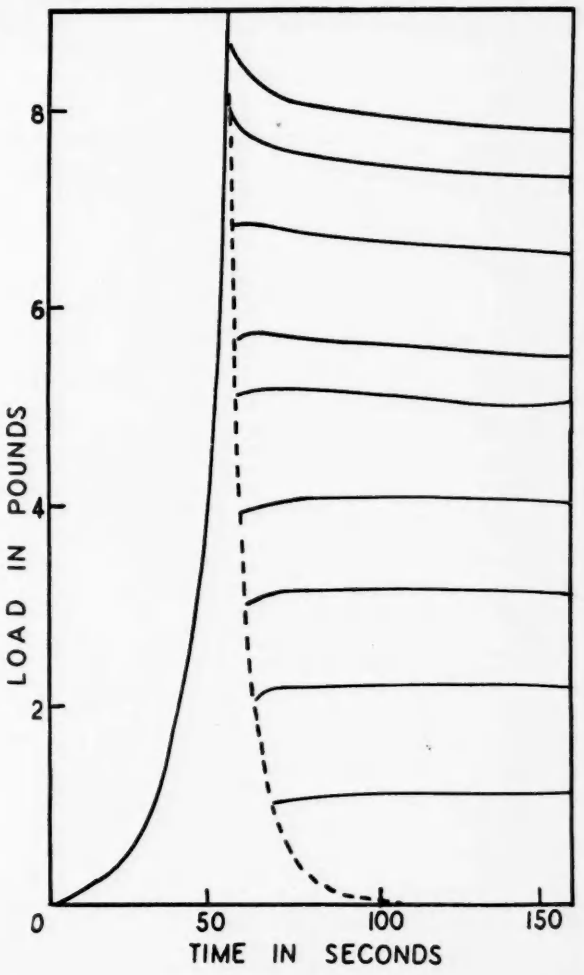

Fig. 6. *Comparison of load-relaxation curves at different recovery loads, plotted in a manner similar to that in Fig. 4, for a blanket fabric.*

ploys a rectilinear chart, thereby providing a means for evaluating the resulting curves in the most simple manner. This is of considerable advantage, as may be seen from the section on interpretation of results. The data may also be analyzed continuously by employing an electronic integration circuit for which provision has been made at the base of the unit described here. This unit has been designed and is being incorporated in the instrument. It permits the energy of compression and of recovery to be indicated directly without the necessity of removing the chart from the recorder and planimetering the areas thus drawn.

The rate at which the chart is moving may also be controlled by change gears, thus providing a means for producing the most easily handled type of trace for a particular specimen subjected to a particular rate of deformation. The chart and the traveling foot move at speeds which are independent of each other but may be related through the gears used in each unit. Thus, time on the chart vertical scale may be converted to penetration of the specimen by noting the dial gage and the particular gears used in each unit. The final graph obtained from the recorder may therefore be considered a load-penetration diagram. If the traveling foot is stopped independently of the chart, and the chart is allowed to continue, the chart vertical scale becomes a time scale related only to its own change gears and the test becomes one of load relaxation at a given penetration.

One further advantage of this type of recorder is that the traveling pen has a high speed of re-

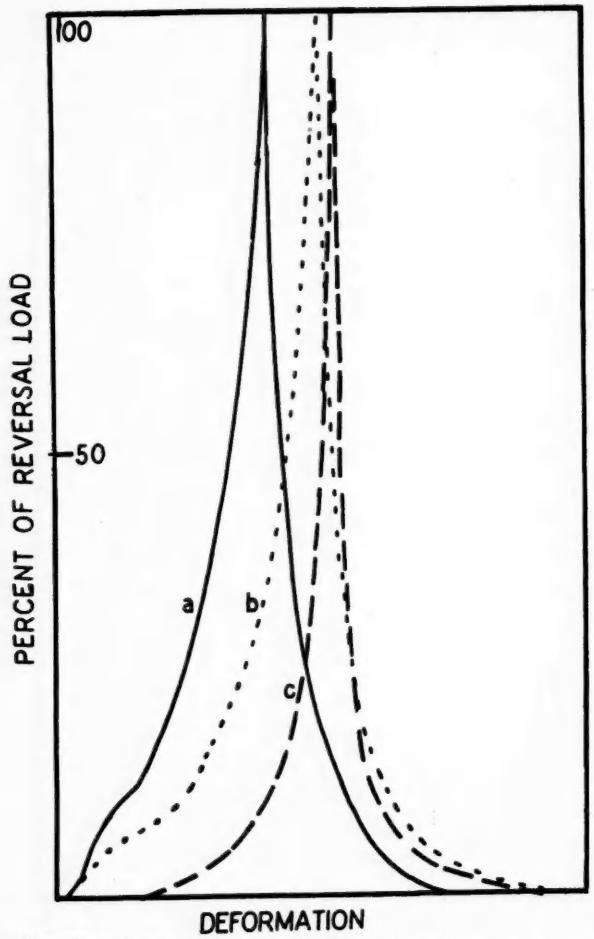

FIG. 7. *Comparison of load-compression curves at different reversal loads for a plush fabric. The curves are plotted in terms of percentage of reversal load, as the instrument actually plots in this manner when a new load scale is selected for the recorder. The characteristic behavior of the pile may be seen to vanish as the load scale is changed. (a)—1-pound reversal load, (b)—2-pound reversal load, and (c)—10-pound reversal load.*

sponse to changes in load applied to the lower head by the specimen. It requires only 1.0 second for the pen to travel the entire length of the chart. Thus, during a particular test at a point of reversal or stoppage of the traveling foot, load changes in the specimen which are large but extend over the space of 0.1 second may be faithfully recorded. This gives a new aspect to such matters as load relaxation and load reversal where such short time phenomena have been hypothecated but never measured accurately owing to the high reversal inertia of recording mechanisms employed by investigators in the past. Some of these results may be seen in the following section.

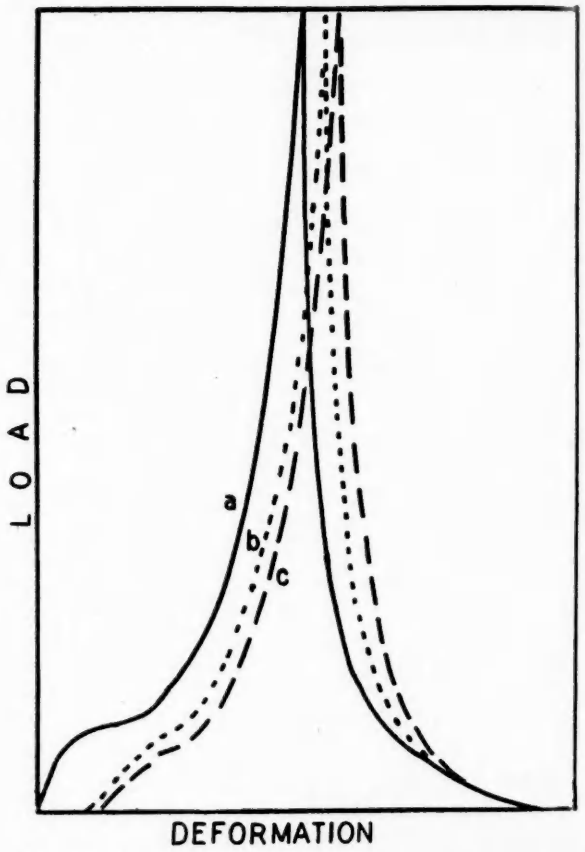

FIG. 8. *Comparison of load-compression curves at different rates of compression for a plush fabric. (a)—0.2 inch per minute, (b)—0.1 inch per minute, and (c)—0.05 inch per minute.*

D. Use of the Instrument and Analysis of Results

1. Compressional Characteristics of Textile Materials

Although the instrument described is a constant rate-of-deformation instrument, the character of the response of the textile material to such deformation is subject to the method by which it is deformed and, consequently, the method in which the instrument is utilized. For example, consider the response curve which results when a material is subjected to a constant rate of deformation until a certain load is reached, the direction of deformation instantaneously reversing at this load, and the material being allowed to recover to zero load at a constant rate. Figure 3 presents such a curve for a blanket fabric. Qualitatively, it may be seen that the material has low resistance to deformation for a considerable depth and then, upon reaching considerable compression, becomes very dense and builds up load rapidly over a small increment of deformation. This is the portion

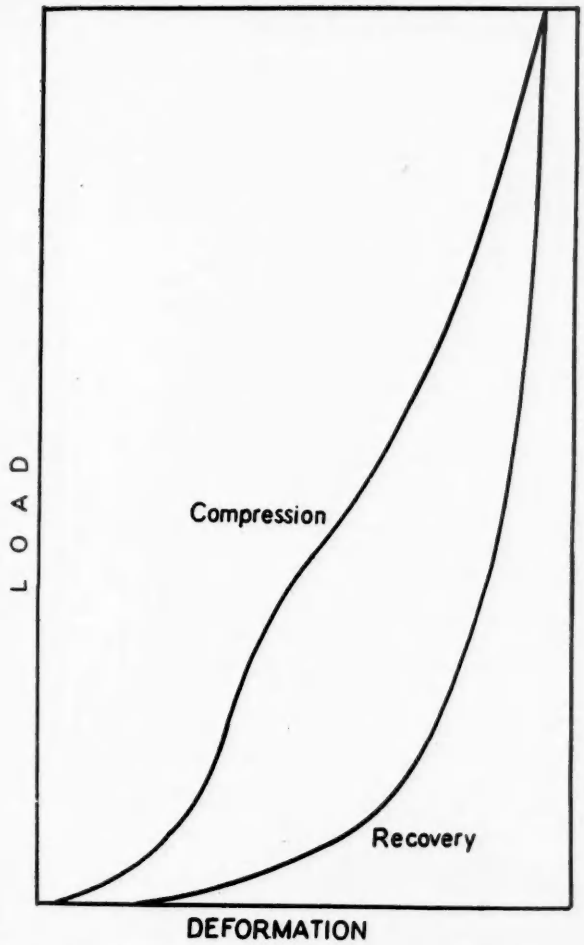

FIG. 9. *Single compression-recovery curve for an Axminster carpet fabric showing the characteristic effect of the compression of the pile.*

marked *a–b*. Upon reaching *b*, the direction of deformation is instantaneously reversed. It may be seen that the recovery rate is much lower owing to the fact that the instantaneous elastic character of the viscoelastic material plus the delayed elasticity of recovery is opposed by the superposition of the delayed elastic component of compression, thus reducing the total effect when compared with the compression cycle. Thus, energy of recovery is less than that of compression by the amount of this memory effect [35].

Some of this memory effect may also be of a semipermanent nature [35] because of the change in mechanical condition brought about by the first compression cycle. Successive cycles reduce this effect until a conditioned cycle is reached, after which further cycles repeat the conditioned cycle without change. Any one of these cycles may then be taken

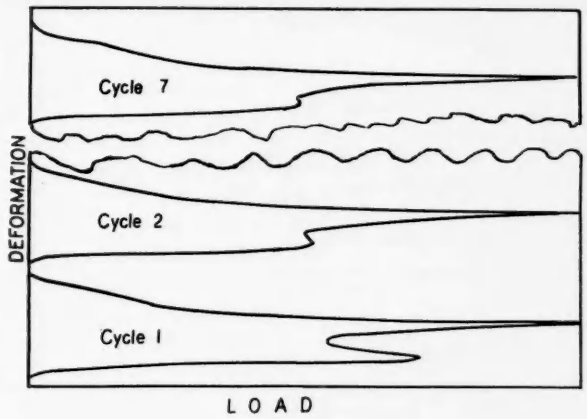

FIG. 10. *Comparison of compression-recovery cycles as they appear on the recorder chart for a mohair pile fabric. The effect of successive cycles on the characteristic performance of the pile is illustrated.*

to represent the working properties of a material under the particular deformation conditions employed in the instrument. Figure 3 also shows the first cycle and one of the conditioned cycles to indicate the energy-of-compression difference described above.

Upon reaching the conditioned cycle, there are several ways in which the instrument may be employed to indicate other elements of viscoelastic behavior. Figure 4 shows the effect of stopping the instrument instantaneously at a given deformation below the maximum reached at *b* (Figure 3) and then allowing the material to undergo stress relaxation at this deformation. If for different places in the fabric the conditioning load (and likewise the time and depth of compression) is changed, then the viscous rate of recovery increases with increasing value of conditioning load. This is again a memory effect since it is proportional to the total time during which the deformation is applied and is thus a function of the delayed elasticity. This behavior is approximately semilogarithmic with time for the period during which it is examined and the character of such relaxation may therefore be given by the semilog time coordinates in Figure 5 with the ordinate plotted as the maximum conditioned load reached. It can be seen from the slopes thus determined that there is apparently a relationship between the conditioning load and the rate of stress relaxation. In fact, in this case there is a direct relationship. If the rate of stress relaxation based on a semilog time scale is divided by the conditioning load, a percentage

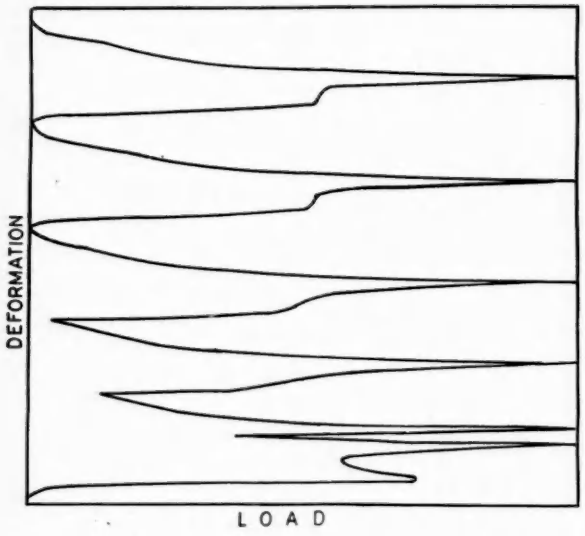

FIG. 11. *Compression-recovery curves as they appear on the recorder chart indicating the characteristics of the compression-recovery cycle for a mohair pile fabric. Various reversal points on the recovery curve are shown in order to indicate the portion of the recovery which governs the return of the pile to the erect condition.*

rate of stress relaxation will result which is virtually independent of the conditioning load. It is not the intent in this paper to determine the exactness of such a relationship, but rather to indicate the type of behavior which may be investigated.*

Quite a different effect is produced when the stress relaxation is obtained after allowing the material to recover to various degrees on the conditioned cycle, as in Figure 6. There are several reasons for this. If the instrument is stopped when the recovery cycle has nearly approached zero load, there is an instantaneous reversal of load with an increase which rapidly reaches an asymptotic load value. During this recovery cycle, the instrument foot is traveling faster with respect to the fabric because of the fabric memory effect previously described. Consequently, upon stopping at the low load, the material builds up its delayed elastic recovery load. The elapsed time is great enough, however, that the total effect is small. It has been shown in Figure 4 that stress relaxation at the maximum conditioned load produces a very pronounced stress-relaxation curve. Between these two limiting cases, the resulting curve is a proportionate combination of the two effects.

* The next paper of this series will deal in more detail with the type of behavior in which the initial relaxation load is obtained practically instantaneously in order to eliminate the possible influence of the time taken to arrive at this load (see footnote, page 165).

The effect of conditioned-cycle rate-of-deformation on such behavior has yet to be investigated.

Various portions of the compression and recovery conditioned cycle may be examined by varying the load scale of the chart. This also varies the depth of compression and the time, proportionately. Superposition of the results obtained for an alpaca plush fabric is given in Figure 7. It can be seen that certain characteristics of behavior may be obscured unless the scales are properly selected.

In addition to the possibility of investigating the response of materials on various load scales, the rate of deformation for any load scale may be varied within the limitations of the instrument. The results obtained at three different deformation rates are given in Figure 8 for the plush fabric. Superposition of the three response curves suggests that distinctive characteristics indicated by such curves are sensitive to the rates of deformation used. This emphasizes the care which must be taken if it is desired to extrapolate the results obtained on this apparatus to a particular service use.

2. Compressional Characteristics of Bulk Fiber

The compressional characteristics of bulk fiber may be investigated in a similar manner. A modified technique must be used, however, to manipulate such materials. It is necessary to confine the material

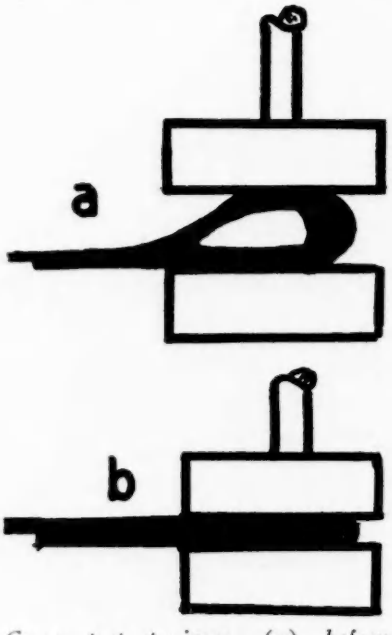

FIG. 12. *Crease test specimen. (a)—before compression; (b)—after compression.*

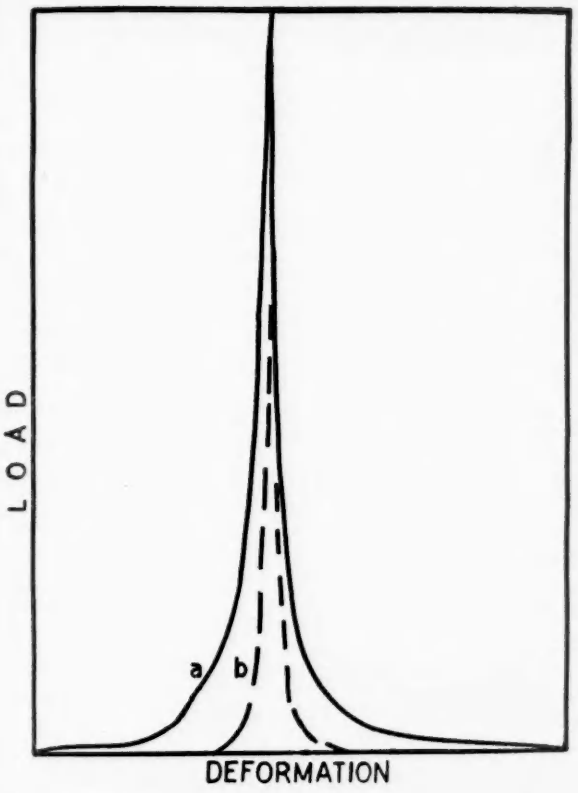

FIG. 13. *Comparison of compression-recovery curve of a folded worsted suiting fabric with that of its double thickness. (a)—compression-recovery curve for the folded fabric; (b)—compression-recovery curve of two thicknesses of the same material. The reversal thickness and load coincide.*

laterally in order to have a material to test. For this purpose, a cup of larger size than the diameter of the deforming foot is employed. Any of the methods described above for obtaining response curves may then be used on this class of materials. Inasmuch as the curves for bulk fiber are, qualitatively, similar to those of fabric, they will not be repeated here. They do, however, show homogeneous behavior over the entire investigational range owing to their geometrical simplicity as a mass. A technique for investigating the stress relaxation after a very high rate of compression will be the subject of the next paper in this series; the apparatus used is an adaptation of the more universal instrument described here to a special class of industrial materials and their application.

3. *Compressional Characteristics of Fabrics*

In the section on the characteristics of textile materials in general, it was indicated that the quantitative behavior of each type of fabric may be obtained from its compression and recovery curves and from its stress-relaxation behavior. It would now be of interest to examine several widely different materials which illustrate the ability of the instrument to respond to the multiphase behavior of some of the geometrically complex structures. The response curve for the blanket fabric shown in Figure 3 illustrates, in the first portion of the curve, the compressional characteristics of the bulk fiber of which the nap is formed. As the material is compressed, the more densely woven structure gradually affects the curve until it becomes the dominant feature on the upper end.

The plush fabric used in obtaining Figure 7 had a particularly high pile composed of relatively fine fibers. Hence it would be expected that the initial resistance to compression would be that of the buckling of the pile, followed by its bending, and then the compression of the bent pile into the fabric. This three-phase behavior is clearly shown in Figure 7 by the various points of inflection or transition zones, primarily on the compression half of the cycle. The pile buckling occurs at such a low load that it was necessary to use the 1-pound scale to show this behavior well. Higher scales gave results in which this behavior was lost in the lower portion of the diagram. The resulting curves were more like those for the blanket material.

An Axminster carpet fabric has a much shorter but a denser pile composed of coarser fibers. Therefore Figure 9 shows a response curve similar, qualitatively, to that of the plush fabric. The transition zone becomes more of a change of inflection in the curve than a true phase-behavior change and also occurs at a much higher load than it does for the plush.

Figure 10 is an actual chart obtained for a mohair upholstery fabric. The pile was not particularly dense but the fibers were very coarse and stiff. The first cycle shows a distinctive behavior pattern in which the pile is initially responsible for the compressional behavior. It acts as if it were a column supported on an elastic foundation [42]. At a very critical value of deformation, the column suddenly buckles and goes into the bending phase. The foundation, on the other hand, relaxes and recovers from the initial heavy load. The pronounced transition from the column phase of behavior to a combination bent-pile and compressed-foundation phase may then be clearly seen. Further cycling gradually reduces this behavior to a minimum but it becomes asymptotic

when fully conditioned. Even at this condition, the two-phase behavior is quite apparent.

The mohair fabric also shows distinctive behavior on the recovery cycle, as seen in Figure 10, At the lower end of the curve is a region of inflection which may be correlated with the return of the pile to the column condition. This is shown more conclusively in Figure 11. The first recovery cycle was reversed at a load value above the point of inflection and a new compression curve was obtained in cycle 2. This curve showed no pile-buckling behavior, indicating that it had not been allowed to return to its initial unbent condition. The second recovery cycle was reversed in the transition region. The third compression curve shows the result of allowing some of the pile to return to its original condition. Complete recovery of the third cycle followed by a fourth complete cycle returns the material to its original conditioned behavior, as may be seen by comparison of Figure 11 with Figure 10.

4. Compressional Characteristics of Creased Fabrics

Many attempts have been made to define crease-resistance of fabrics, but the chief difficulty in arriving at a satisfactory definition has been the inability to measure the behavior. The instrument described herein has been applied to such behavior with the intent of developing a technique for investigating such behavior. It is hoped that this may ultimately result in an accepted approach to the evaluation of crease-resistance.

The most satisfactory method of handling a fabric specimen is to cut a specimen 1½ inches wide of sufficient length that when folded back upon itself the fabric ends lie flat on each other. In this condition it is inserted under the foot of the instrument in such a manner that, when fully compressed, the folded edge remains under the deforming foot. Upon reaching this condition, the position of the fabric under the foot is carefully determined for later use. These two extreme conditions are indicated schematically in Figure 12. Such a creasing and uncreasing cycle is shown in Figure 13 for a worsted suiting fabric. The folded fabric is then removed and reversed so that a double thickness of the fabric is inserted under the foot in the same amount as was the compressed folded fabric. A cycle is then obtained for the double fabric. The results of these two cycles are indicated by superposition in Figure 13. It may be seen that the area between the two curves represents the characteristics of the material due only to its creasability.

Acknowledgment

The author wishes to express his appreciation for the work done in the preparation of the section on the design and construction of the instrument by Mr. G. S. Parsons, instrumentation engineer of the M.I.T. Textile Division, and for work done in obtaining the curves on the instrument by Mr. A. M. Spiro, part-time technician with the M.I.T. Slater Memorial Research Laboratory. Special appreciation is expressed to Miss Ruth L. Freedman, laboratory technician of M.I.T. Textile Division, for her painstaking work in preparing the curves for publication.

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Thixotropic Viscoelastic Systems

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Contribution of the Textile Foundation and Princeton University, Frick Chemical Laboratory, Princeton, New Jersey

Abstract

An attempt is made to explain the thixotropic behavior of certain fibers when they are stretched by postulating a number of potential energy surfaces which may govern the nature of the viscous flow within the fiber. On the basis of these energy surfaces, equations are obtained relating rate of elongation and applied stress, and these equations are integrated numerically to give a relationship between applied stress and actual elongation. The energy surface giving results which approach the results of actual experiment is a simple modification of the surface which Eyring used to explain shear flow in viscous materials [3].

IN OBSERVING the stress-strain characteristics of fibers in this laboratory, pecularities have been noticed which have been attributed to thixotropic behavior of the fiber. After one or two cycles of a reloading experiment, the stress-strain curve of a fiber frequently has the form shown in Figure 1, where the solid line represents observed behavior and the broken line represents "normal" or "idealized" behavior. The thixotropic behavior is seen to be a resistance to plastic flow (the region of the curve where departure from the initial steep slope occurs is the region of plastic flow) greater than that which is normally offered and a subsequent breakdown of that resistance with the result that steady flow finally occurs again at almost normal impressed force. This paper reports an investigation of the possibility of explaining such a phenomenon by considering the potential energy surface of the system and treating the problem as one of reaction rates [1].

Let us consider the "three-element model" (Figure 2) which was investigated by Halsey, White, and Eyring [2] and which explains the general behavior of fibers subjected to stretching. The total stress on the model is the sum of two components: one a linear function of the elongation, contributed by the single (open) spring, and the other a component contributed by the spring and dashpot in series,

$$F = k_2 l + f. \tag{1}$$

We shall now resolve the stress-elongation curve of Figure 1 into these two components and plot the force f on the dashpot and spring in series against the total elongation. The f vs. l curve for the second elongation is shown in Figure 3, where again the dotted curve represents idealized or normal behavior and the full curve the observed behavior. It can be seen from Figure 2 that the rate of elongation of the viscous portion of the spring-dashpot combination is given by

$$\frac{dl_1}{dt} = \rho - \frac{1}{k_1}\frac{df}{dt}, \tag{2}$$

where

$\rho = \dfrac{dl}{dt}$ = over-all rate of elongation of the model,

= constant, in the experiments which have

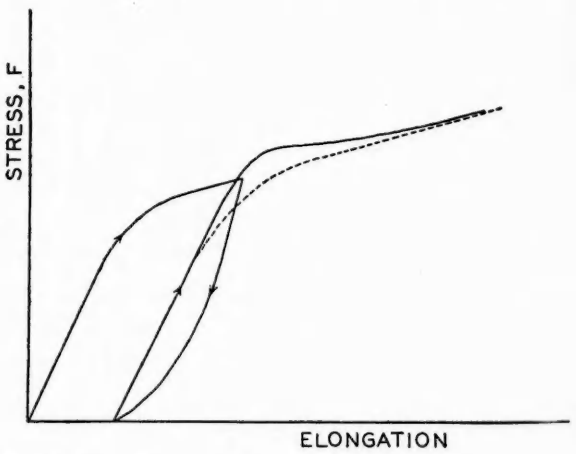

FIG. 1. *Type of stress-strain curve that shows thixotropic behavior.*

– – – – – *Curve in which thixotropic behavior is not shown.*

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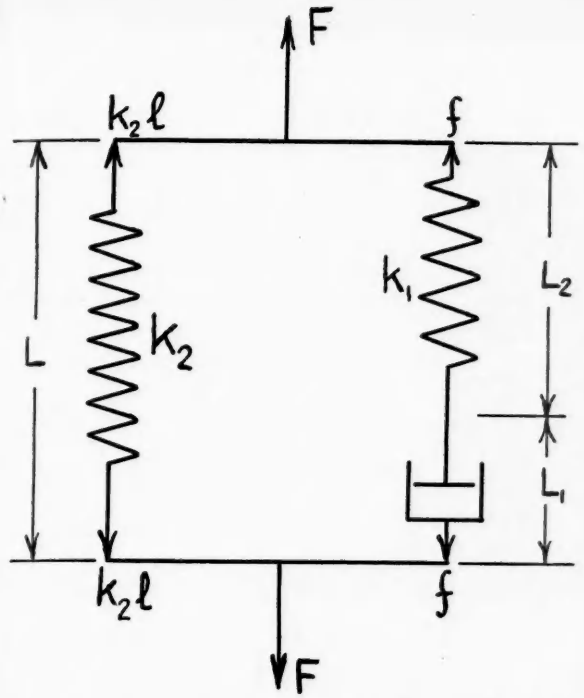


FIG. 2. *The three-element model. Strain associated with L is l; L_1 is l_1; and L_2 is f/k_1.*

been performed in this laboratory, and

k_1 = force constant for the spring in series with the dashpot.

Equation (2) will be combined with other expressions for $\frac{dl_1}{dt}$, obtained by considering potential surfaces which may represent the action of the viscous units—that is, the dashpots or flow units in a group of models. The surfaces to be used are of the nature of that set up by Eyring in his analysis of shear flow in viscous liquids [3], pictured in Figure 4. Here the valleys are successive positions of equilibrium along the direction of force, corresponding to the packing of the molecules of the liquid in such a way that their energy is low. When flow occurs, the change in configuration between one equilibrium position and the next involves a temporary configuration of higher free energy. Therefore the flow process will have an activation free energy that corresponds to the excess energy of this transition state. As far as simple shear is concerned, there is no need to distinguish successive equilibrium positions because flow rates are identical whichever position is considered.

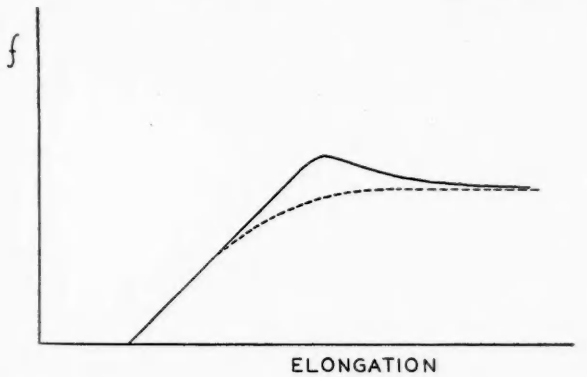


FIG. 3. *Stress on the series combination of spring and dashpot vs. elongation, showing thixotropic behavior.*
– – – – – *Curve in which thixotropic behavior is not shown.*

The first surface is depicted schematically in Figure 5, where each circle represents an equilibrium position for a flow unit, the positions of state A being at uniformly lower energy than those of state B. The population of all the equilibrium positions in state A is represented by a fraction n of the total number of flow units, and the population of state B by $(1 - n)$. Positions in state B are reached by flow over a potential barrier from state A when tension is applied to the viscoelastic material. Flow between positions in state B corresponds exactly to Eyring's viscous flow for liquids in simple shear [3]. The ease of flow between positions in state B is characterized by the constant K, and by DK for flow between A and B, where D is an arbitrary constant. If $D < 1$, flow from A to B is more difficult than flow between positions in B, and consequently the potential barriers between A and B are higher than those between positions in B.

Following the method that Halsey, White, and Eyring used [2] to show that, for a flowing dashpot,

$$\frac{dl_1}{dt} = K \sinh \alpha f$$

which, when αf is large,

$$\cong \frac{Ke^{\alpha f}}{2},$$

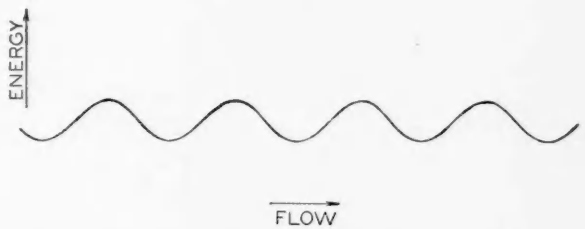


FIG. 4. *The potential energy surface for Eyring shear flow.*

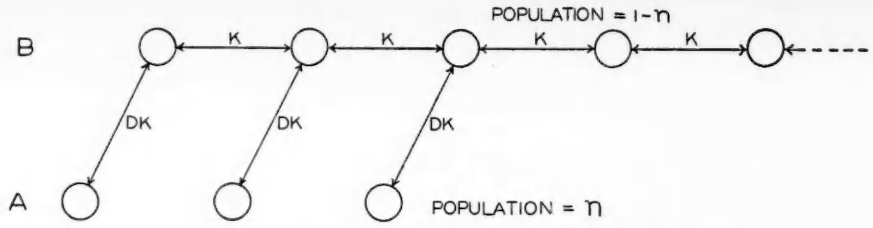


FIG. 5. *Schematic representation of Eyring shear model having continued series of equilibrium positions.*

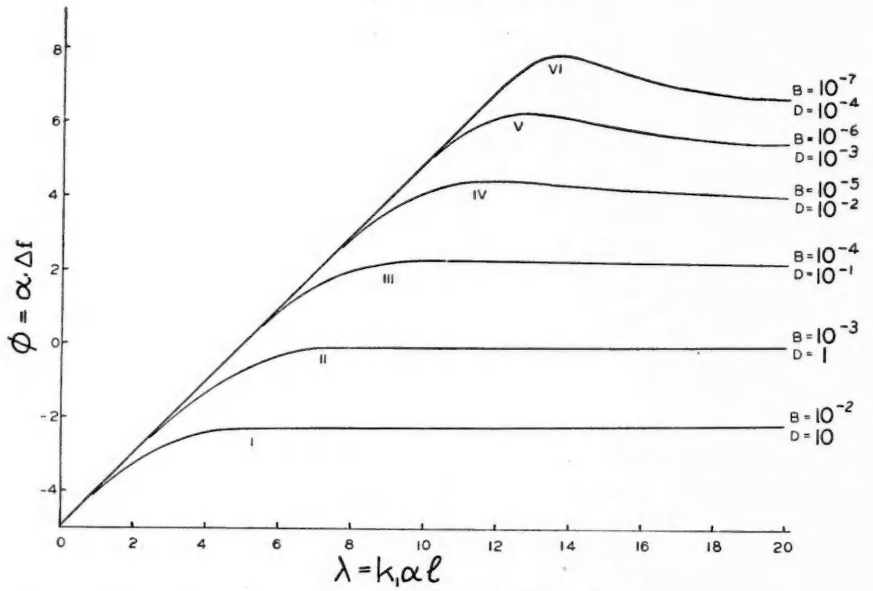


FIG. 6. *Relationship between ϕ and λ when flow in the higher energy level follows Eyring's model of continued equilibrium positions for flow of liquids.*

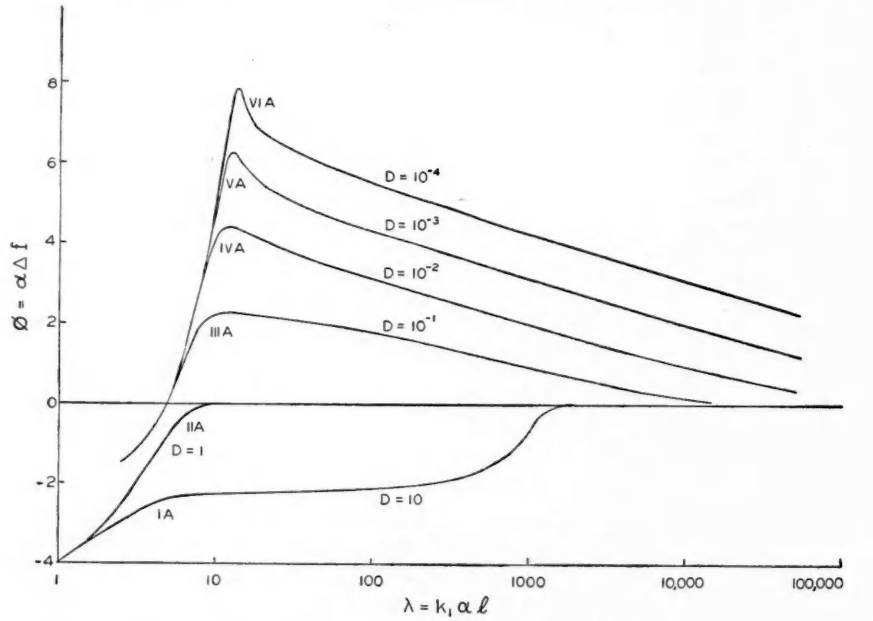


FIG. 7. *Relationship between ϕ and λ when flow in the higher energy level follows Eyring's model of continued equilibrium positions, showing the logarithmic decay of the thixotropic force Δf as stretching is continued.*

we find that the rate of elongation as a result of flow from A to B is $\frac{nDK}{2} e^{\alpha f}$ and the rate of elongation as a result of flow between positions in state B is $\frac{(1-n)K}{2} e^{\alpha f}$ (see Figure 5). The approximation that $\frac{dl_1}{dt} \cong \frac{K}{2} e^{\alpha f}$ is justified in this work because αf is large in the region where thixotropic phenomena occur. Hence we obtain

$$\frac{dl_1}{dt} = \tfrac{1}{2}(1-n)Ke^{\alpha f} + \tfrac{1}{2}nDKe^{\alpha f}. \tag{3}$$

Combining equations (2) and (3),

$$\rho - \frac{1}{k_1}\frac{df}{dt} = \tfrac{1}{2}(1-n+nD)Ke^{\alpha f}. \tag{4}$$

Setting $f = f_\infty + \Delta f$ and substituting $\frac{\rho}{K} = \beta$, then

$$\beta - \frac{1}{Kk_1}\frac{d\Delta f}{dt} = \tfrac{1}{2}(1-n+nD)e^{\alpha f_\infty}e^{\alpha \Delta f}. \tag{5}$$

Now $\alpha f_\infty \cong \ln 2\beta$. Setting $\phi = \alpha \Delta f$ and noting that $\rho dt = dl$, equation (5) can be reduced to

$$1 - \frac{1}{\alpha k_1}\frac{d\phi}{dl} = (1-n+nD)e^{\phi}$$

or

$$1 - \frac{d\phi}{d\lambda} = (1-n+nD)e^{\phi}, \tag{6}$$

where $\lambda = \alpha k_1 l$.

The rate at which the population of state A changes is

$$\frac{dn}{dt} = -n\frac{DK}{2}e^{\alpha f},$$

and by making the appropriate substitutions we can reduce this equation to

$$\frac{dn}{d\lambda} = -nBe^{\phi}, \tag{7}$$

where

$$B = \frac{D}{\alpha k_1}.$$

Convenient values for α and k_1 are $\alpha = 4 \times 10^{-9}$ cm^2/dyne [2] and $k_1 = 2.5 \times 10^{11}$ dynes/cm^2 [4]; then $B = 10^{-3}\ D$. After arbitrarily selecting a value of D, one can solve equations (6) and (7) simultaneously by point-by-point integration. When λ is sufficiently large, the approximation that the rate of elongation of the Maxwell unit equals the rate of viscous flow can be used to simplify the calculations—that is, when

$$\rho = \frac{dl_1}{dt},$$

then equation (4) reduces to

$$\rho = \tfrac{1}{2}(1-n+nD)Ke^{\alpha f},$$

whence

$$e^{-\phi} = 1 - n + nD.$$

Substituting this in equation (7) gives

$$\frac{dn}{d\lambda} = -\frac{nB}{1-n+nD}.$$

Integrating,

$$\ln n - (1-D)n = -B\lambda + \text{constant}. \tag{8}$$

For the special case of $D = 1$,

$$1 - \frac{d\phi}{d\lambda} = e^{\phi}$$

and

$$\lambda = \ln\left(\frac{\text{const.}\ e^{\phi}}{1-e^{\phi}}\right). \tag{9}$$

The equations have been integrated for $D = 10$, 1, 10^{-1}, 10^{-2}, 10^{-3}, 10^{-4}. For the point-by-point integration, equations (6) and (7) were put in the form

$$\Delta\phi = \{1 - [1 - n(1-D)]e^{\phi}\}\Delta\lambda$$
$$\Delta n = -nBe^{\phi}\Delta\lambda,$$

from which $\Delta\phi$ and Δn are calculated for an arbitrarily selected value of $\Delta\lambda$. Initial conditions were taken as $\lambda = 0$, $n = 1$, and $\phi = -5$. If the condition $\phi = -5$ when $\lambda = 0$ should not represent reality, the origin of λ may be moved as far as required without changing the values of ϕ. The results of the integrations are assembled in curves I to VI of Figure 6, and in curves IA to VIA of Figure 7, which show logarithmic decay of the thixotropic force Δf with continued elongation. The change in the population of the lower energy state A as the viscoelastic material is elongated is shown in the plots of n against λ in Figure 8.

The slow logarithmic decay of thixotropic force found in the curves of Figure 7 is not shown by the thixotropic materials that have been studied in this laboratory. It is at this point that the first essential difference between the shear model and the viscoelastic model is observed. The reason for this slow decay of stress is the fact that once a small fraction of the flow unit population reaches the

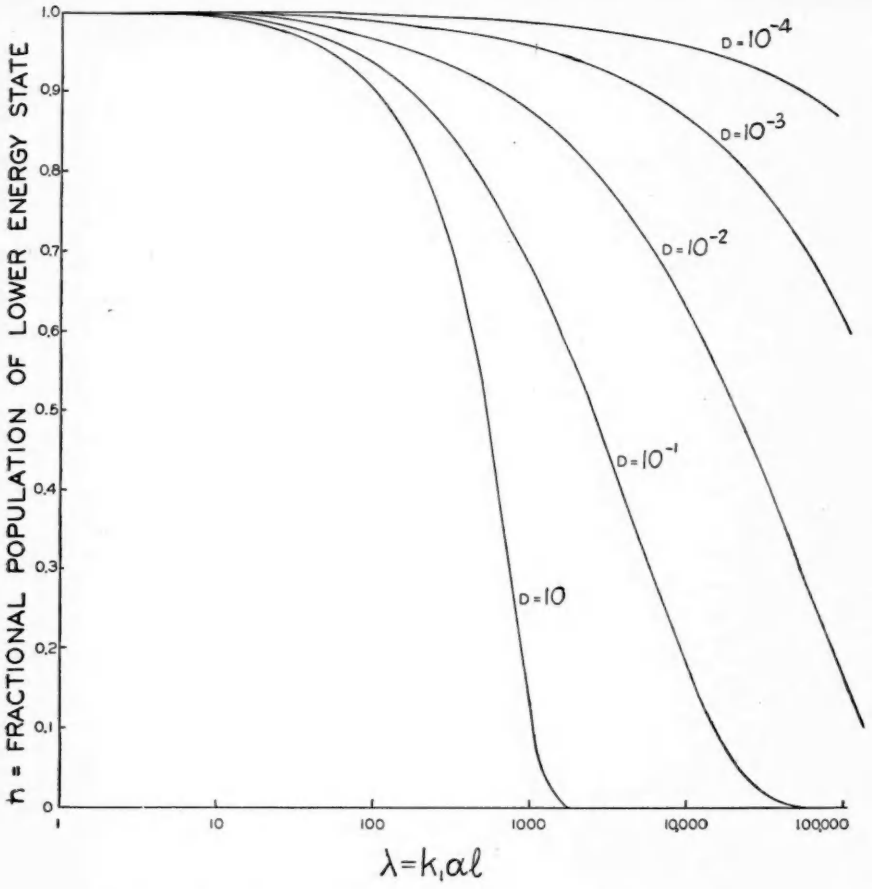
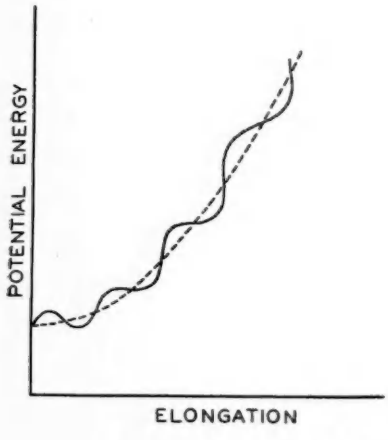


FIG. 8. *Variation of the population of the lower energy state with elongation of the flowing system for the Eyring model of continued equilibrium positions.*

upper energy level in which flow is easy, this small fraction does all the flowing that is required to keep up with the forced elongation of the material and the lower energy state is not swept out by a momentarily increased force, *f*.

The next model for consideration is one in which successive equilibrium positions in the higher energy state *B* are placed on a parabolic potential energy surface which is such that flow requires the population to climb up the side of the parabola (see Figure 9a). This corresponds to the incorporation of an elastic character into the dashpot action since here we have superimposed the potential energy surface of a simple shear unit on the parabolic potential energy surface of a spring. In this model, the lower state *A* should be swept out more rapidly than in the case considered previously. As indicated in Figure 9b, the suc-



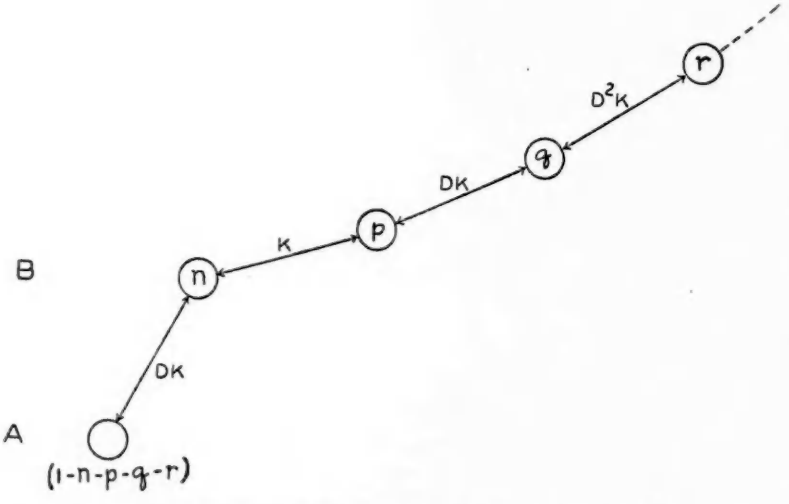


FIG. 9a (*left*). *Potential energy surface for elastic character in conjunction with flowing dashpot.*

FIG. 9b (*right*). *Schematic representation of "parabolic model."*

cessive equilibrium positions are separated by successively higher potential barriers across which the ease of flow is characterized by the constants K, DK, D^2K, etc.; and the populations of successive positions are n, p, q, r, etc. Four of these positions are considered here so that the population of state A is $1 - n - p - q - r$. Ease of flow from state A to the first level of state B is characterized by the constant DK. By the same method as was used before, the rate of elongation due to flow out of state A is $\frac{1}{2}(1 - n - p - q - r)DKe^{\alpha f}$, and the rate of flow due to flow from the first position of state B to the second is $\frac{1}{2}nKe^{\alpha f}$, etc. For all positions considered:

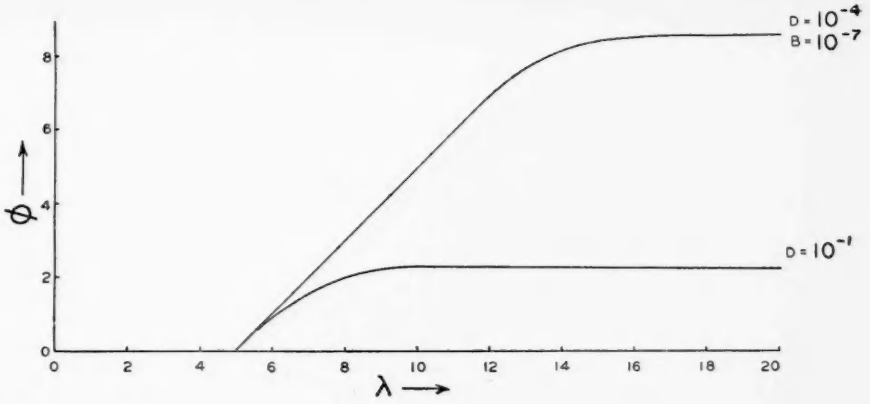


FIG. 10. *Change of ϕ with λ for a model in which equilibrium positions in the higher energy level are arranged on a parabolic surface.*

$$\frac{dl_1}{dt} = \tfrac{1}{2}(1 - n - p - q - r)DKe^{\alpha f} + \tfrac{1}{2}nKe^{\alpha f} + \tfrac{1}{2}pDKe^{\alpha f} + \tfrac{1}{2}qD^2Ke^{\alpha f};$$

also (equation (2)):

$$\frac{dl_1}{dt} = \rho - \frac{1}{k_1}\frac{df}{dt}.$$

Using the same substitutions and simplifications as before, we obtain:

$$1 - \frac{d\phi}{d\lambda} = [D + n - nD - qD + qD^2 - rD]e^{\phi}; \quad (10)$$

also

$$\frac{dn}{d\lambda} = \left((1 - n - p - q - r)B - \frac{nB}{D}\right)e^{\phi}, \quad (11)$$

$$\frac{dp}{d\lambda} = \left(\frac{nB}{D} - pB\right)e^{\phi}, \quad (12)$$

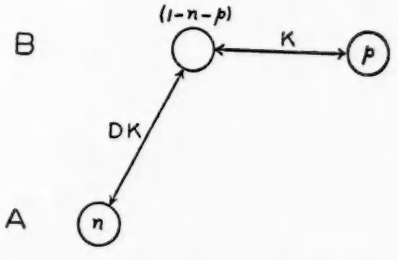


FIG. 11. *Schematic representation of a model which has only three equilibrium positions.*

$$\frac{dq}{d\lambda} = [pB - qBD]e^{\phi}, \quad (13)$$

$$\frac{dr}{d\lambda} = qBDe^{\phi}. \quad (14)$$

Equations (10) to (14) may be solved simultaneously by point-by-point integration. Since no useful approximations have been found for use when λ is large, the solution of these equations has not been completed. The change of ϕ with λ is plotted in Figure 10 as far as it has been calculated and the populations are shown in Table I. For $D = 10^{-1}$, and at low values of λ where the thixotropic hump appears, there is virtually no difference between the behavior of this model and of the simple model. As D becomes smaller, however, the sides of the parabolic energy surface become steeper, and flow

TABLE I. POPULATIONS OF THE SEVERAL EQUILIBRIUM POSITIONS IN THE "PARABOLIC MODEL"

	λ	n	p	q	r
$D = 10^{-1}$	5.0	0	0	0	0
	6.0	1.3×10^{-4}	7.8×10^{-8}	0	0
	8.0	1.0×10^{-3}	6.0×10^{-6}	1.0×10^{-10}	0
	10.0	2.7×10^{-3}	3.5×10^{-5}	5.0×10^{-7}	1.0×10^{-10}
	12.0	5.6×10^{-3}	9.4×10^{-5}	5.0×10^{-6}	3.0×10^{-9}
	14.0	6.4×10^{-3}	2.0×10^{-4}	1.0×10^{-5}	8.0×10^{-9}
	16.0	8.1×10^{-3}	3.3×10^{-4}	1.5×10^{-5}	1.5×10^{-8}
	18.0	9.8×10^{-3}	4.9×10^{-4}	2.0×10^{-5}	2.2×10^{-8}
	20.0	11.4×10^{-3}	6.8×10^{-4}	2.4×10^{-5}	2.7×10^{-8}
$D = 10^{-4}$	5.0	0	0	0	
	10.0	3.3×10^{-6}	9.0×10^{-9}	0	
	12.0	5.0×10^{-5}	7.3×10^{-6}	0	
	14.0	1.0×10^{-4}	3.5×10^{-4}	6.5×10^{-8}	
	16.0	1.0×10^{-4}	1.2×10^{-3}	6.1×10^{-7}	
	18.0	1.0×10^{-4}	2.1×10^{-3}	2.0×10^{-6}	
	20.0	1.0×10^{-4}	3.1×10^{-3}	4.4×10^{-6}	

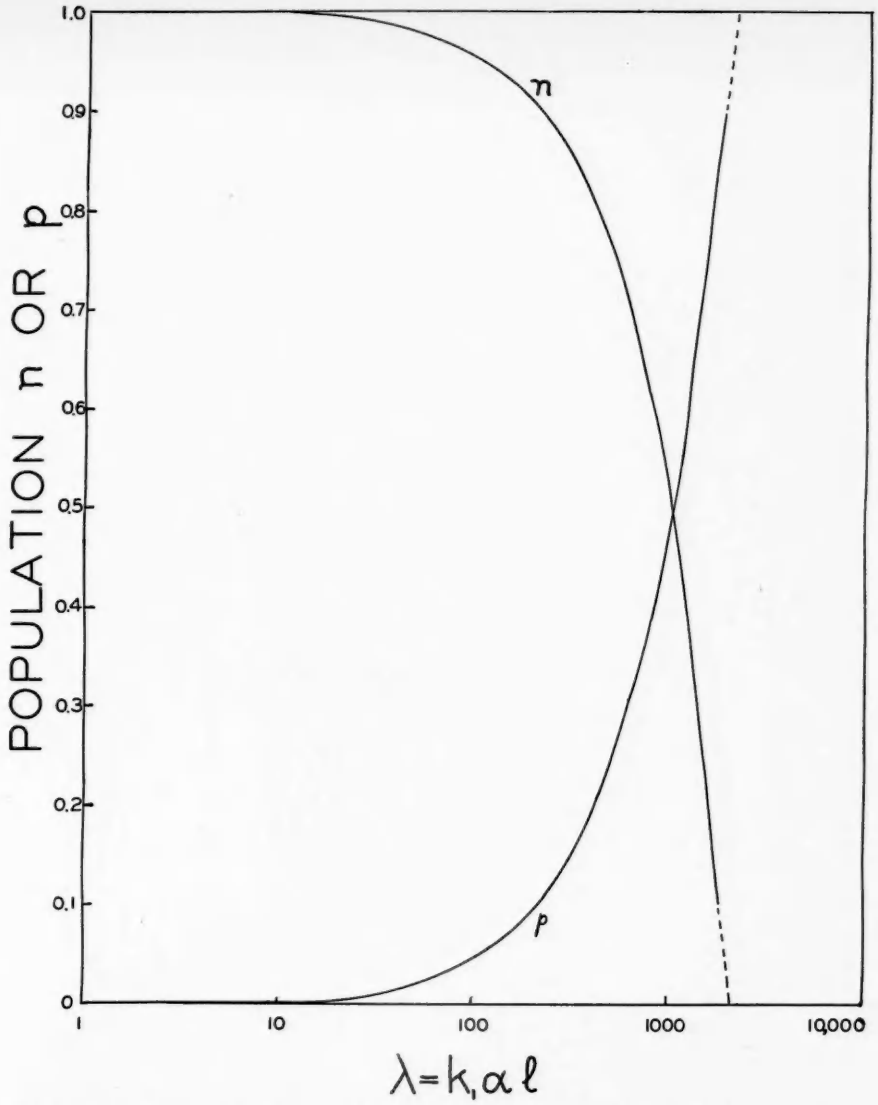


FIG. 14. *The populations of the initial (n) and final (p) equilibrium positions for a model having only three equilibrium positions; the curves shown are for* $D = 10^{-3}$ *and these are not distinguishable from curves for* $D = 10^{-4}$ *on the scale used.*

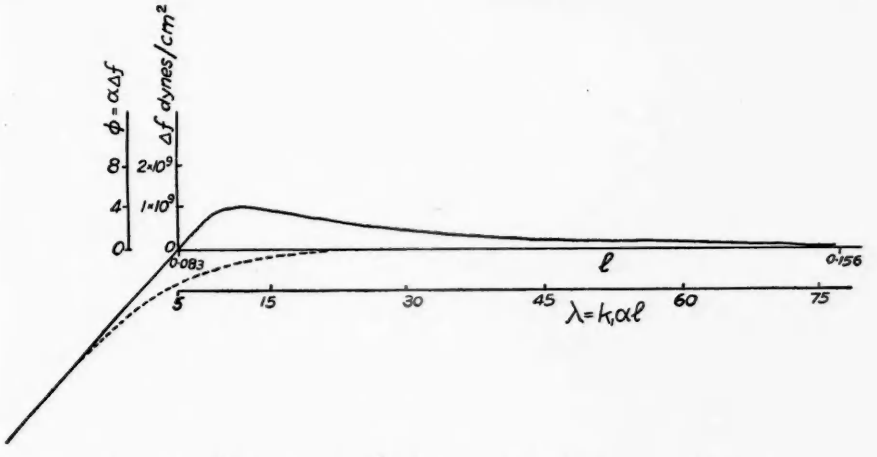


FIG. 15. *Experimental* ϕ *vs.* λ *curve for a degummed silk fiber.*
– – – – – *Curve in which thixotropic behavior is not shown.*

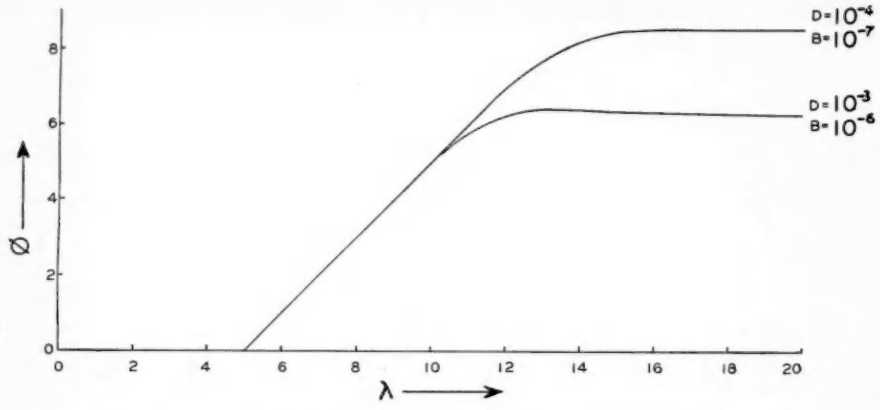


FIG. 12. *Change of ϕ with λ for a model in which there are only two equilibrium positions in the higher energy level.*

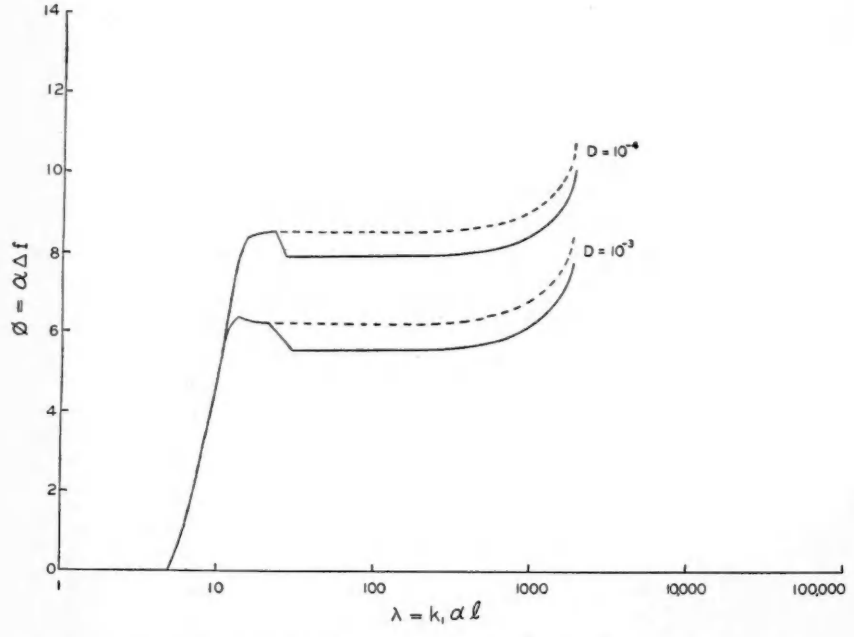


FIG. 13. *Showing variation of ϕ with λ for a model having only three equilibrium positions.*

——— *Curve calculated for larger values of λ by available approximation equations.*

– – – – – *Curve for larger values of λ arbitrarily corrected by adding 0.65 to all values of ϕ calculated by the approximation equations.*

beyond the second equilibrium position of state B becomes progressively more difficult. When $D = 10^{-4}$, flow beyond the second position of state B is so difficult that no decrease in elongating force is observed on stretching—that is, the thixotropic hump vanishes.

A model for which the calculations are simpler is one in which there are only two equilibrium positions in the upper energy level, B. This may be considered to be a degenerate case of the "parabolic model" in which the sides of the parabola quickly become infinitely steep. The populations of the three positions and the nature of the potential barriers between positions are indicated in Figure 11. The equations governing the behavior are

$$\frac{dl_1}{dt} = \rho - \frac{1}{k_1}\frac{df}{dt} = \tfrac{1}{2}(1-n-p+nD)Ke^{\alpha f}, \quad (15)$$

$$\frac{dn}{dt} = -\tfrac{1}{2}nDKe^{\alpha f}, \quad (16)$$

and

$$\frac{dp}{dt} = \tfrac{1}{2}(1-n-p)Ke^{\alpha f}, \quad (17)$$

from which we can get, by the methods used before,

$$1 - \frac{d\phi}{d\lambda} = (1-n-p+nD)e^{\phi}, \quad (18)$$

$$\frac{dn}{d\lambda} = -nBe^{\phi}, \quad (19)$$

$$\frac{dp}{d\lambda} = (1-n-p)\frac{B}{D}e^{\phi}. \quad (20)$$

Equations (18), (19), and (20) may be solved simultaneously by point-by-point integration. It is observed, when performing this integration, that $\frac{dp}{d\lambda} \cong -\frac{dn}{d\lambda}$ for larger values of λ. With this approximation, equations (19) and (20) may be combined to give $1 - n - p = nD$; whence, from equation (15),

$$\frac{dl_1}{dt} = (1-n-p)Ke^{\alpha f}.$$

Assuming also that $\rho \cong \frac{dl_1}{dt}$, then

$$e^{-\phi} = 2(1-n-p). \quad (21)$$

ABSTRACTS

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A list of periodicals abstracted, the abbreviations used in references to articles, and the addresses of publishers of English language publications is given in the January, 1948, issue, page 48.

ANALYSIS: TESTING: LABORATORY METHODS

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Fiber Gage

Air gage measures fiber fineness. W. S. Smith. *Textile Inds.* **111**, 86–8 (Nov. 1947).

A description of an air gage that is used to measure the fineness of textile fibers. The gage is based upon the principle that air permeability or porosity is inversely proportional to fiber fineness. Air is drawn through a plug of fibers and the resistance to the flow is measured. The resistance increases as the fineness of the fibers increases. Fine fiber stock offers more numerous air passages but the openings between the fibers are smaller. Duplicate tests made on the gage seldom differ more than 0.2 μg. per in. on cotton and the difference is usually less than 0.1 μg. Inaccurate weighing and preparation of the sample are the two chief causes of the discrepancies. Trash or foreign matter will tend to increase the porosity whereas dust will give the opposite effect. A. L. Landau

Text. Research J. Mar. 1948

Fiber Testing

Fiber testing aids the cotton classer. Seth Carter. *Textile World* **97**, 110–11, 216, 218 (Nov. 1947).

The summary of a survey of 336 mills by the U. S. Dept. of Agriculture with the purpose of listing the methods and practices of testing the cotton bought by the mills for specific purposes. Fiber strength was considered to be the most important fiber property affecting yarn quality. Fiber length and uniformity, fiber fineness, and fiber maturity were considered to be of lesser importance, in the order named. The importance of variety and the place of growth are factors affecting spinning quality which are becoming more generally recognized. Economy of processing was deemed more vital to the mill than the economy of purchasing the raw material. Laboratory testing of the fibers proved valuable as a supplementary source of information to indicate the potential spinning quality of the cotton bought by the mill. However, the regular cotton-classing procedure is still the standard means of judging the cotton, as laboratory testing is too slow to allow the testing of each bale of a lot. The results of laboratory tests and the grading of the cotton classer may be coordinated to determine the exact value of the fiber. Four tables are given showing the comparisons that should be made between laboratory tests and the cotton classers' estimates. The four values to be determined are length uniformity, fiber strength, fineness, and the amount of impurities present. A. L. Landau

Text. Research J. Mar. 1948

Particle-Size Determination

Latex particle sizes as determined by soap titration and light-scattering. H. B. Klevens. *J. Colloid Sci.* **2**, 365–74 (June 1947).

Particle sizes of synthetic latices (isoprene–styrene), determined by titration using several dyes as indicators of surface saturation, agree with values obtained optically. To obtain concordant results, it was necessary to free the system of unreacted monomer. E. D. Klug

Text. Research J. Mar. 1948

Sampling and Testing Methods

Testing as an aid to efficiency in finishing. E. G. H. Carter. *J.*

Combining equations (20) and (21),

$$p = \frac{B}{2D}\lambda + \text{constant.} \qquad (22)$$

Also,

$$\frac{dp}{dn} = \frac{p + n - 1}{2nD}$$

which, on integration, gives

$$p = \frac{n}{2D - 1} + 1 + \text{constant } n^{1/2D}. \qquad (23)$$

Equations (21), (22), and (23) may then be used as an approximate solution until ϕ starts to increase at such a rate that $\rho \cong \frac{dl_1}{dt}$ is no longer valid. It is easy to show that $\phi = \lambda + \text{constant}$ when λ is very large. From equations (18) and (20),

$$d(\lambda - \phi) = \left[\frac{D}{B} + \frac{nD^2}{B(1 - n - p)}\right] dp.$$

When λ is large, $dp = 0$; hence $\lambda - \phi = \text{constant}$. The changes in ϕ and in population with elongation, λ, are shown in Figures 12, 13, and 14. It is apparent from Figure 13 that the approximation equations (21), (22), and (23) are not entirely satisfactory, and the values of ϕ calculated by these equations have been arbitrarily increased to what appears to be more reasonable values by adding 0.65.

It is interesting to compare the theoretical curves with some experimental results. Figure 15 is a plot of the excess "thixotropic stress," Δf, against elongation, l; or, as shown by the second set of axes, of ϕ vs. λ, taken from a stress-strain curve for degummed Japanese silk run in this laboratory. The values of k_1 and α used to calculated ϕ and λ from Δf and l were taken as 2.5×10^{11} dynes/cm^2 and 4×10^{-9} cm^2/dyne, respectively, which were the same values used in calculating the theoretical curves. The λ axis was arbitrarily displaced from $\lambda = 83$ at $\phi = 0$ to $\lambda = 5$ at $\phi = 0$ to correspond to the calculated curves. The curve in Figure 15 resembles most the curves in Figure 6 except for the fact that the excess stress, Δf, does not decay fast enough in the calculated curves.

Some degree of success has thus been attained in explaining the thixotropic behavior of a fiber by assuming that Eyring shear flow occurs in a higher energy region which is reached by flow of the viscous element over a relatively high potential barrier.

Acknowledgment

The authors wish to thank Dr. J. H. Dillon, under whose direction the work described in this paper was done, for reading the manuscript and clarifying the presentation; and Miss Corneil Messler for preparing the figures and the typescript.

Literature Cited

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4. Stein, Richard, Halsey, George, and Eyring, Henry, Mechanical Properties of Textiles, IV, TEXTILE RESEARCH JOURNAL **16**, 53 (1946).

Soc. Dyers and Colourists **63**, 337–9 (Nov. 1947).

A discussion of the value of general testing and sampling methods is followed by the development of an analysis of a sampling method used in testing the felting shrinkage of chlorinated wool fabrics.

J. A. Woodruff

Text. Research J. Mar. 1948

Titration of Surface-Active Agents

Volumetric analysis of colloidal electrolytes by turbidity titration. Joseph M. Lambert. *J. Colloid Sci.* **2**, 479–93 (Nov. 1947).

The concentration of surface-active agents is determined by a turbidimetric titration of an anionic agent with a cationic agent or vice versa, under such conditions that a colloidal precipitate is produced near the equivalence point and is solubilized or coagulated by an excess of reagent. Titration data on 12 different surface-active agents are given. For example, Igepon T is titrated with cetyl pyridinium chloride.

E. D. Klug

Text. Research J. Mar. 1948

CHEMICAL AND PHYSICAL RESEARCH

★

Alpha-Cellulose

"Noble" cellulose and α-cellulose. Germano Centola. *Ind. carta* (Milan) **1**, 39–47 (1947) (through *Chem. Abstr.* **41**, 7743*i* (Nov. 20, 1947)).

A cellulose called "noble" has a high content of α-cellulose (*I*)—i.e., the fraction insoluble, under standard conditions, in a 17.5% NaOH solution—but the opinion that a cellulose of a good reactivity should contain high amounts of *I* is correct only if relating to an ideal type of *I*. The actual molecules of technical *I* are different from the ideal by certain properties which are not measured in the ordinary analytical examination. These properties depend principally on transverse bonds between the macromolecules, made with bridges of primary valencies, perpendicular to the fiber axis. To define a "noble" or a highly reactive cellulose, it is necessary to prepare the fractionation diagram (*cf.* Sieber, *Die chem.-technischen Untersuchungsmeth. der Zellstoff u. Papierind.*, Berlin, 1943, p. 544), and to calculate from this diagram the "polymolecularity" (*P*) so that the celluloses, with respect to their reactivity, are classified as: (1) good, containing 10–20% of $P < 150$, 45–60% $P = 150$ to 1,000, and 25–40% $P > 1{,}000$; and (2) bad, containing 10–25% $P < 150$, 20–35% $P = 150$ to 1,000, and 50–60% $P > 1{,}000$. The values may be variable, according to the chemical treatments made to prepare celluloses for different purposes.

Text. Research J. Mar. 1948

Colors and Absorption Spectra

The limiting colors due to ideal absorption and transmission bands. I. H. Godlove. *J. Optical Soc. Am.* **37**, 778–91 (1947).

Synthetic-organic chemists have developed to a high state the art of prediction of changes in absorption spectra with changes in molecular architecture but they are not able to visualize the colors resulting from specific changes. The colorimetrist is frequently asked what spectra constitute ideal goals at which to aim in a particular field of colors. In this issue of the *J. Optical Soc. Am.*, the author presents calculations for colors produced by idealized (rectangular) absorption and transmission bands of various wave lengths from 30 mμ to 300 mμ and expresses the results in terms of I.C.I. and Munsell specifications, giving also the name of the nearest TCCA standard sample. Since Munsell chromas for the calculated bands and for the nearest TCCA standards are given on each side of the name, it is easy to see how close—or distant, if that is one's point of view—chemists now are to limit colors. The author points out that no true greens can be made with a single absorption band, and no magentas with a single transmission band. Saturation of dyes increases with width of absorption or narrowness of transmission bands, but a "law of diminishing returns" operates, there being a point beyond which it is unprofitable to go. For example, a 5-mμ shift in the yellow has more effect on the color than a 50-mμ shift in the blue-green. In two spectral-transmission regions, it makes little difference whether very wide or very narrow bands are used; in others the reverse is true. Saturation maxima were found at 3 absorption wave-length positions, and for transmission at 400–450 mμ. Many previous correlations of spectra and "color" are invalidated because of the "crowding" of some hues into narrow wave-length regions and great spread in others. In practice, yellows approach closest to the ideal maximum possible saturation; greens are far away. The spectral conditions for better greens are outlined. Figures are included that show both the Munsell chroma and the Munsell value obtained versus the wave-length center of the bands, also I.C.I. diagrams showing the color gamut for each band width calculated. This paper should be of vital interest to every textile chemist and colorist, and will give to all others interested in textile coloring problems a down-to-earth picture of what the limiting colors are. The points lie on the same surface as points found by MacAdam, but the Godlove series is of more direct interest to chemists since it is worked out in terms of tools already used by them in studying "color" and chemical structure.

D. Nickerson

Text. Research J. Mar. 1948

Structure and Properties of Elastomers

Preparation and properties of rubberlike high polymers. IV. Correlation between structure and properties of elastomers derived from dienes. G. Salomon and C. Koningsberger. *J. Polymer Sci.* **2**, 522–41 (Oct. 1947).

The correlation between structure and properties of elastomers pre-

pared from dienes, natural rubber, and gutta-percha, as well as those of significant derivatives of natural rubber, is discussed. The similar influence exerted by methyl groups on the brittle point, elastic recovery, and permeability to gases is demonstrated and separated from the effect caused by the insertion of —CH_2—CHR— groups in straight-chain polybutadiene, which takes place during copolymerization or 1,2 addition. The preponderant influence of an unbranched-chain structure on tensile strength at elevated temperatures and in the swollen state is illustrated. Complex compounds with silver nitrate provide further evidence for the particular symmetry of the natural rubber chain. They are reinforcing elements for synthetic elastomers. The brittle points of hydrochlorides from natural rubber, gutta-percha, and polyisoprene are compared with those of polyvinyl compounds. Correlation between the structure of molecular units in a polymeric paraffin derivative and its elasticity is indicated. Author

Text. Research J. Mar. 1948

Electrophoresis

Electrophoresis in an ultracentrifugal field. E. Reed Lang, Quentin Van Winkle, and Wesley G. France. *J. Colloid Sci.* **2**, 315–31 (June 1947).

An apparatus designed for the investigation of electrophoresis of colloidal systems in an ultracentrifugal field was constructed and the electrophoretic mobilities of ferrous oxide sols were determined. The particle size or sedimentation constant of hydrous ferric oxide sols increases with continuous exposure to the combined effect of an electrical and ultracentrifugal field. Low concentrations of potassium citrate lower the mobility of ferric oxide sols and increase the sedimentation constant and particle size.

Text. Research J. Mar. 1948 E. D. Klug

Light-Scattering

A note on the symbols and definitions involved in light-scattering equations. Victor K. La Mer and Marion D. Barnes. *J. Colloid Sci.* **2**, 361–3 (June 1947).

A convenient set of symbols for use in light-scattering equations is recommended with the objective of eliminating the confusion in the literature. E. D. Klug

Text. Research J. Mar. 1948

Membrane Behavior of Keratin

Membrane potentials for keratin. S. Baxter. *J. Colloid Sci.* **2**, 495–508 (Nov. 1947).

Measurement of the membrane potentials of keratin membranes have been made using KCl and NaCl; the results show how the selectivity of the membranes can be altered by soaking in suitable buffer solutions. The results support Torrell's theory of membrane behavior in dilute salts, and some membrane potential measurements using HCl solutions are compared with Gilbert and Rideal's predictions. A qualitative theory is suggested for the mobility-pH curves obtained by electrophoresis. It also explains why the isoelectric point of keratin is not at a pH of 7, which is the pH of zone-membrane selectivity. Author

Text. Research J. Mar. 1948

Degradation of Polymers

Some structural and chemical aspects of ageing and degradation of vinyl and diene polymers. Robert B. Mesrobian and Arthur V. Tobolsky. *J. Polymer Sci.* **2**, 463–87 (Oct. 1947).

Solutions of styrene and polystyrene in toluene were refluxed and small amounts of benzoyl peroxide were added periodically. Relative viscosities of both sets of solutions approached the same value—a steady state for a given set of conditions attained both by polymerization and depolymerization. Data showing the influences of light, oxygen, and photosensitizers on rates are given. It is assumed that similar reaction mechanisms are involved in polymerization and in ageing. The component reactions in ageing are depolymerization and aggregation, which includes cross-linking. To isolate these reactions, studies were made of the sol-gel properties—i.e., the amount of gelation, the swelling index, and the change in viscosity of the sol of coagulated GR-S and Hevea latices with and without inhibitors. In general, inhibitors retarded gelation and viscosity drop. Polystyrene, polymethyl methacrylate, and polyisobutylene films, heated at 100°C, 131°C, and 150°C, lost viscosity without gelation. Permanent-set data were obtained for several rubbers by measuring intermittent and continuous stress relaxations. Oxygen-absorption-rate data at 130°C are given for various rubber polymers, rubber stocks, and plastics, together with similar data obtained when inhibitors were present. The effect of compounding and vulcanization was also studied.

Text. Research J. Mar. 1948 E. D. Klug

Permeability of Polymers

Permeability of polymers by gases and liquids. R. Houwink. *Verfkroniek* **20**, 172–6 (1947) (through *Chem. Abstr.* **41**, 7812*g* (Nov. 20, 1947)).

The influence of entropy and internal energy on the permeability of phenol-formaldehyde resins, polyethylene, polyvinyl chloride, polystyrene, polymethyl methacrylate, polyamides, polyvinyl acetate, cellulose acetate-butyrate, cellulose tri-acetate, ethylcellulose, cellulose acetate-propionate, cellulose acetate. The effect of the temperature (positive or negative) on their solubility depends on the shape of the potential curve between the penetrating molecules and the high-polymer molecules. It is probable that the attraction between these molecules determines the phenomena observed. The diffusion through rubberlike polymers must always be of the activated type, whereas for hard polymers it can be activated, partly activated, or not activated, depending upon the existence of attracting centers on the molecules. The voids in cellulose are considered essentially different from those in phenol-formaldehyde resins, expressed by diverging diffu-

sion. The addition of plasticizers does not greatly change the permeation and not much difference is observed in the permeability of the polymers by liquids in comparison to that by gases. 17 references, 3 tables.

Text. Research J. Mar. 1948

Tensile Properties of Rubber

Tensile strength of pure-gum natural rubber compounds. Geoffrey Gee. *J. Polymer Sci.* **2**, 451–62 (Oct. 1947).

The tensile properties of pure-gum natural rubbers are reviewed. It is shown that the tensile-strength measurements depend on the amount of crystallization at break. Cross-linking affects tensile strength in that it prevents plastic flow and permits crystallization during stretching. A highly cross-linked rubber is weak because rupture occurs before elongation is great enough to permit crystallization. The chemical reactions in vulcanization inhibit crystallization by producing structural modifications. The phenomena of overcure, reversion, and ageing are discussed.

Text. Research J. Mar. 1948 E. D. Klug

Thermal Conductivity

Water content of a thin film of regenerated cellulose and its thermal conductivity. Chujiro Matano. *J. Phys.-Math. Soc. Japan* **16**, 222–3 (1942) (through *Chem. Abstr.* **41**, 7744*d* (Nov. 20, 1947)).

The thermal condition of cellophane is proportional to water content up to 15%, and then increases linearly with water content. The break in the curve at the water content of 15% shows a change in the state of adsorption of water molecules.

Text. Research J. Mar. 1948

Soap in Organic Solvents

Behavior of some alkali soap systems of organic solvents. Mata Prasad, G. S. Hattiangdi, and B. K. Wagle. *J. Colloid Sci.* **2**, 467–77 (Nov. 1947).

The solubility of sodium oleate, sodium stearate, and sodium palmitate in a large variety of solvents was determined. Although solubility was poor at room temperature, clear, mobile solutions were obtained near the boiling point of the solvent. On cooling, the soap remained in solution, crystallized out, or formed a gel, depending on the solvent. The conditions which give rise to true gels and pseudo gels were studied. E. D. Klug

Text. Research J. Mar. 1948

Viscosity-Molecular Weight Relationship

Intrinsic viscosity-molecular weight relationship for polystyrene. A. I. Goldberg, W. P. Hohenstein, and H. Mark. *J. Polymer Sci.* **2**, 503–10 (1947).

A polystyrene polymer of $[\eta]$ in toluene of 2.08 was prepared by emulsion polymerization at 63°C. This polymer was carefully fractionated by solvent volatilization and the intrinsic viscosities and osmotic molecular weights of fractions were determined in toluene and butanone. The equation $[\eta] = 3.7 \times 10^{-4} M^{0.62}$ for toluene and $[\eta] = 7.0 \times 10^{-4} M^{0.53}$ for butanone best expressed the results. A table based on data in the literature gives constants for the above equations for several cellulose derivatives and synthetic polymers.

Text. Research J. Mar. 1948 E. D. Klug

BLEACHING: DYEING: FINISHING

★

Stripping with Hydrosulfites

The stripping of dyed textiles by the use of hydrosulfite compounds. J. Starkie. *J. Soc. Dyers and Colourists* **63**, 340–3 (Nov. 1947).

A detailed résumé is given of the stripping of wool with sodium hydrosulfite, basic zinc sulfoxylate-formaldehyde, sodium sulfoxylate-formaldehyde, and neutral zinc sulfoxylate-formaldehyde. Procedures are also given for the stripping of vat- and azoic-dyed cotton and viscose rayon. Some results on the stripping of dispersed acetate dyes from nylon are described.

J. A. Woodruff

Text. Research J. Mar. 1948

Wool Dyeing

Theory and practice in wool dyeing. F. L. Goodall. *Am. Dyestuff Reptr.* **36**, P380–5 (July 14, 1947).

Based on the assumption that swelling is the governing factor in the dependence of the dyeing on temperature, a new technique for applying "colloidal" dyes (e.g., Polar Yellow R) was developed. The customary dyeing method was altered in three respects, as follows: the goods were introduced into the bath as near the time it reached the boiling point as possible, Glauber salt was omitted, and the initial acid content of the bath was considerably reduced. At the boiling point fiber pores are largest, the dye is more nearly molecularly dispersed, and therefore uniform distribution throughout the fiber is more readily obtained. Glauber salt, on the other hand, tends to restrict swelling and to increase dye-particle size. In the absence of Glauber salt, initial dye absorption is controlled by regulating the acidity of the dye bath. Colloidal dyes applied in this way have been found to produce more level, brighter shades of superior fastness to rubbing and with an extremely high degree of penetration. K. S. Campbell

Text. Research J. Mar. 1948

Detergents

Theory and practice of textile-processing agents. A. Chwala and A. Martina. *Textil-Rundschau* **2**, 147–61 (1947).

A comprehensive survey of ionic and other soaplike auxiliaries useful in textile operations. Molecular structures serve as the basis for the classifications. Classical and novel mechanisms of solvation in aqueous

baths are shown in detail in diagrams. The relationship of constitution to colloidal behavior and characteristic properties of 17 distinct groups of auxiliaries are described. Specific practical applications are given for many derivatives, together with a list of 117 (mostly European) commercial products. A general view of interrelations among the major groupings of fatty derivatives, whatever the mechanism of aqueous solvation, yields some interesting rules for wetting agents, detergents, and surface-active fatty materials. J. B. Eisen

Text. Research J. Mar. 1948

Enzymes

Enzymes in textile processing. P. J. Wood. *Am. Dyestuff Reptr.* **36**, P355–61 (June 30, 1947).

A review of the production and application of the enzymes used in textile processing. Four methods for the evaluation of enzymes are given in detail. K. S. Campbell

Text. Research J. Mar. 1948

Textile Resins

Textile resins. W. H. Jaeger. *Am. Dystuff Reptr.* **36,** P352–4 (June 30, 1947).

A review of the application of urea-formaldehyde and methylated melamine resins to fabric for the production of a variety of permanent finishes, pigment dyeing and printing, and wool-shrinkage control. K. S. Campbell

Text. Research J. Mar. 1948

Rayon Slashing

"Monofilizing"—main aim of rayon slashing. Anon. *Textile World* **97**, 115, 186, 188, 190, 192 (Oct. 1947).

This is an abstract from a book soon to be published entitled *Rayon Technology*. A well-slashed continuous-filament viscose rayon warp can be woven at an operating efficiency of 95%. Cellulose-base yarns swell in cross section, lengthen, and lose approximately 45% of their strength when wet. Viscose will recover most of this stretch upon drying; acetate will not. Stretch caused by the slashing process should be limited to 6% for viscose, 3% to 4% for most other types of rayon. Low-twist acetates are most difficult to slash and usually gain about 6% to 7% stretch. Overheating of the warp to attain faster drying will cause a high loss of fiber resiliency. Viscose rayon should be dried to approximately 10% moisture regain. A. L. Landau

Text. Research J. Mar. 1948

Wool-Shrinkage Control

Wool made shrink-resistant by improved chlorination. L. Shapiro. *Textile World* **97**, 151, 153, 210, 212, 214, 216 (Nov. 1947).

Two new processes for controlling the shrinkage of wool without damaging the fiber by using wet chlorination have been introduced. One method makes use of alkaline solutions, the other of acid. The alkaline process imparts a very soft hand, excellent strength, abrasion resistance, and level dyeing properties to the wool at low cost. The acid process has the added advantage of producing very white wool without further bleaching but is more expensive. The application of the solutions may be made at any stage of processing the fibers, usually just before dyeing. The chemical reactions that take place in producing and stabilizing the chlorinating bath are fully explained, as is the full procedure to be followed in using either type of bath. A. L. Landau

Text. Research J. Mar. 1948

FIBERS: YARNS: FABRICS: MECHANICAL PROCESSES

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Fiber Surface

Specific surface and electrokinetic potentials of cellulose fibers. S. G. Mason. *Pulp & Paper Mag. Can.* **48**, 76–80 (1947) (through *Chem. Abstr.* **41**, 7747*g* (Nov. 20, 1947)).

A brief review is given of the measurement of the specific external surface area and the surface potentials of cellulosic fiber suspensions; representative results are discussed. Of the existing methods for measuring surface area, the permeability method is considered the most promising. Although many of the phenomena involved in fiber flocculation, size precipitation, filler retention, pitch control, etc., are often explained on the basis of changes in the zeta-potential, measurements of this quantity have not been sufficiently extensive to establish conclusively the validity of this type of reasoning. Future work is outlined.

Text. Research J. Mar. 1948

Wet Strength of Fibers

Attempts to increase the wet strength of artificial fibers. Antal Lintner. *Kém. Lapja* **5**, 25–7, 41–3 (1944) (through *Chem. Abstr.* **41**, 7746*b* (Nov. 20, 1947)).

In tests to determine the value of urea-formaldehyde on viscose fibers, large variability was observed in the results. More satisfactory results were obtained when the impregnated fibers were treated with an ammoniacal bath containing a softening agent (fat-alcohol sulfonate); a bath of pH 4 is best. The impregnated fibers should be dried at 60–70°C and then at 110°C for 10 min. A slight excess of the urea-formaldehyde resin appears to increase the wet tearing strength. In washing experiments, no significant loss of the urea resin was observed in 12 washings with Na_2CO_3 and soap.

Text. Research J. Mar. 1948

Mold Growth on Cotton

Factors affecting the growth of mold on cotton fabrics and related materials. W. I. Illman and M. W. Weatherburn. *Am. Dyestuff Reptr.* **36**, 343–4, 369–72 (June 30, 1947).

Study of the influence of tempera-

ture and humidity conditions on the growth of mold on a variety of substances indicated that growth increased in severity and occurred over a wider range of temperatures with rise in humidity up to 100%, and that growth demonstrated an optimal temperature range at each humidity. There was no growth at 60% R.H. and only slight growth at 70% R.H. (86°F). Growth on cotton duck, both untreated and copper-naphthenate-treated, was accelerated by the presence of free moisture. The removal of constituents of unbleached duck by leaching in water decreased the extent of rotting, whereas leaching with solvent had no effect. The addition of various nutrient solutions to the water-leached fabric did not promote growth to the same extent as it did on the untreated fabric. Authors

Text. Research J. Mar. 1948

Kenaf

Kenaf—Fiber plant rival of jute. Julian C. Crane. *Econ. Botany* **1**, 334–50 (July–Sept. 1947).

Kenaf (*Hibiscus cannabinus L.*) has been grown for many years in India, Java, and other Asian and African countries. Investigations during the war in United States and Canada indicated that Kenaf gave greater yields than other soft fibers studied. However, it has been referred to erroneously as "roselle." Requirements with respect to climate, soil, and culture are given. Fibers are from 5 to 10 ft. long. From 1,000 to 3,000 lbs. of fiber and a ton of seed are produced per acre. Kenaf fiber may be employed in the manufacture of gunny cloth, ropes, and cordage, fishing nets, floor mattings, and rug backing. Kenaf seed yields up to 20% oil which, when refined, may be used for salads and cooking purposes. R. B. Evans

Text. Research J. Mar. 1948

Worsted Fabric Calculations

"Ready Reckoner" to ascertain weights. G. Feher. *Text. J. Australia* **22**, 447–8 (Aug. 20, 1947).

A table is presented which is designed for ascertaining weights of cloth (made with worsted yarns) per sq. yd. It can be used (1) to ascertain the weight of cloth from a given thread count with given yarns, (2) to find the thread count necessary to obtain a certain weight of cloth with given yarns, and (3) to find the counts of yarn required to obtain a given weight with a given thread count. The number of ends and picks per in. in finished cloth is expressed in 2 formulas. L. A. Fiori

Text. Research J. Mar. 1948

Tire Cord

Practical problems in the manufacture and service of tires built with continuous filament cord. J. H. Dillon. *Am. Dyestuff Reptr.* **36**, P385–90 (July 14, 1947).

A description of the manufacturing steps in the production of tires, and a discussion of the practical problems involved in tire manufacture and service. These include temperature and moisture relations, adhesion of fiber to rubber, cord strength, stretch and creep, and methods of testing under static and dynamic conditions. Ten desirable characteristics for a tire cord are listed and the several types of tire cord (viz., low-stretch cotton, viscose rayon, hot-stretched nylon, glass, and brass-plated steel) are compared with respect to these properties. In spite of the low wet strength of rayon, which makes tire-processing complicated, better dry strength, impact strength, fatigue life, and somewhat lower temperature coefficient of strength of this fiber outweigh its lower flexibility and higher creep, so that rayon is preferred to cotton for truck tires. The high temperature coefficient of strength of nylon has prevented its use to any extent in truck tires. It is stated that if the cost of steel tire cord could be reduced to a reasonable level it would undoubtedly replace rayon in most truck tires and perhaps later in passenger-car tires. Glass-filament cord is also potentially important in the tire-cord field.

K. S. Campbell

Text. Research J. Mar. 1948

Card Improvements

Tomorrow's card—What will it be? Anon. *Am. Wool and Cotton Reptr.* **61**, 9, 10, 53–54 (Nov. 13, 1947).

The development of the modern card is traced. The various changes in design which have been attempted as a means of increasing either the production of the card or the quality of the sliver are described. Among these are the continuous stripper, 45-in. (width) card, cards with double doffers, and different positions and uses of fancy rolls.

Text. Research J. Mar. 1948 A. L. Landau

Waste Removal in Woolen Carding

Waste thread removal on woolen cards. Anon. *Text. Mercury and Argus* **117**, 652–3 (Nov. 7, 1947); *Platts Bulletin* **5**, 174–6 (July–Aug. 1947).

The best method suggested is to remove the waste ends from the tape before it has passed through the rubbing aprons. This new system of the pneumatic type is built as an integral part of the condenser and operates through an individual fan unit having a tube with ventholes over the waste tapes. The waste is conveyed to a centrally located receptacle. Principal advantages to be gained are a reduction of work load for the operator, a reduction of waste in spinning, elimination of prolonged stoppages in carding, and longer life of the rubbing aprons. L. A. Fiori

Text. Research J. Mar. 1948

Oiling of Cotton

Oiling of cotton. Anon. *Text. Weekly* **40**, 600 (Oct. 3, 1947).

Successful results have been obtained in condenser spinning by oiling cotton with a solution having

a highly purified mineral base combined with a special emulsifier and water. It is a very stable oil emulsion. Marked improvements are noticed in several directions, one of the most important being the reduction of "fly" in the card room. Extraneous matter is more readily separated, resulting in a cleaner sliver. Spinning is improved to the extent that the yarn sheds less and causes fewer ends down. No difficulties are encountered in bleaching and dyeing, as the oil, on first contact with water, immediately washes out completely and may act as a mild detergent to improve quality further. L. A. Fiori

Text. Research J. Mar. 1948

Drawing-Frame Condenser

Waste prevention at the drawing frame. Anon. *Text. Mercury and Argus* **117**, 728 (Nov. 21, 1947); *Platts Bulletin* **5**, 166–7 (July–Aug. 1947).

The Duple condenser for condensing drawing-frame sliver is of particular importance at the finishing head of drawing frames. It increases the amount of sliver that can be held in a can, gives the sliver a smoother and more compact form for subsequent processing, and permits the removal of waste with a minimum of effort and loss of time. The device is relatively simple, consisting of a trumpet-container section which fits into the calender roller cover plate in the usual manner and a trumpet condenser, containing a compressor spring, which fits inside the container. A choke induced by a bad piecing or a thick place in the sliver causes the compression spring to yield, allowing the condenser section to be pulled downward until it contacts the calender roller and activates the electric stop motion. With a slight modification, this apparatus can be applied to drawing frames equipped with mechanical stop motions. L. A. Fiori

Text. Research J. Mar. 1948

Ends-Down Tests

Ends-down tests reveal spinning troubles. F. H. Gunther and Marcus Gross. *Textile World* **97**, 112–13 (Dec. 1947).

Charting the number of ends down on the spinning frame is one of the best methods of determining the cause of inefficiency in the spinning mill. The test will give a clear, concise tabulation of the various reasons for the breakage of ends on the spinning frame. A sample test sheet is shown. A. L. Landau

Text. Research J. Mar. 1948

Cotton Spinning Mills

The ideal cotton spinning mill. W. A. Hunter and W. H. Watson. *Text. Weekly* **40**, 698, 700, 702, 704 (Oct. 17, 1947).

Two ideal 40,000-spindle spinning plants, one producing 30s yarn from American cotton and the other 60s yarn from combed Egyptian cotton, are described in detail in this report. Hank rovings, doublings, drafts, speeds, turns-per-inch, and machine details are all condensed in tabulated form. L. A. Fiori

Text. Research J. Mar. 1948

Nylon Staple

Spinning of nylon staple fibers on the cotton system. John L. Beacon. *Rayon Text. Mo.* **28**, 73–5 (Oct. 1947).

1.5 den., $1\frac{9}{16}$-in. staple is recommended for spinning 100% nylon or blends on the cotton system. In most mills 2 processes of roving are used: in the first the machine is equipped with a long-draft system and in the second it has a conventional drafting system consisting of 3 sets of top and bottom rolls. A system of long-draft spinning is also recommended. The double-belt and single-belt systems are both satisfactory and will produce fine yarns from the shorter staple lengths of nylon. For staple lengths of over $1\frac{9}{16}$ in. a more specialized form of drafting system employing very efficient and flexible drafting zones is used. The adaptability of this special system to staple lengths from $1\frac{1}{8}$ in. to 3 in. makes its use desirable. For medium counts a front roll speed of between 550 to 585 in. per min. is recommended. There seems to be a marked tendency for nylon to cockle while being spun if the roll settings are too close, particularly in the spinning of the finer numbers of yarn from nylon of fine denier. Generally, when processing 100% nylon, 55% to 65% R.H. and 75°F are satisfactory. For blends, conditions are determined to accomodate the other fibers of the mixture. L. A. Fiori

Text. Research J. Mar. 1948

Rayon on Worsted System

Rayon staple spins well on worsted system. Anon. *Textile World* **97**, 129, 131, 196, 198, 200 (Oct. 1947).

Condensed from the book *Rayon Technology*, based on research by the American Viscose Corp. Rayon may be processed on either the Bradford-length or the varied-length worsted system. The fact that rayon staple must be handled more gently than woolen staple is stressed. The mechanical adjustments and changes that must be made are described. A. L. Landau

Text. Research J. Mar. 1948

Nelson Continuous Rayon Process

Nelson continuous rayon process. Anon. *Am. Wool and Cotton Reptr.* **61**, 15–16 (Dec. 11, 1947).

A discussion of the Nelson process of producing rayon, enumerating the advantages of the yarn produced by this method over that formed by the centrifugal or spool-spun method. It is claimed that as the yarn is not touched during the entire process all of the potential dangers involved in handling while it is in a tender stage are eliminated. There are fewer breakages in the filaments of the finer yarn. The mechanics of the process are fully described. A. L. Landau

Text. Research J. Mar. 1948

Rayon Staple Spinning

The influence of rayon staple on preparing and spinning machinery developments. H. Ashton. *J. Text. Inst.* **38**, P340–52 (Aug. 1947).

This report is confined to viscose staple, to (1) the influence of its staple length on yarn strength, (2) the influence of its filament denier on yarn strength, and (3) the effect of variations of staple length and filament denier on the range of yarn counts it is possible to spin in a given material. In addition, a study was made of various spinning systems (cotton, silk, worsted, flax, jute) to discover where changes are needed. So far, the standard staple supplied for cotton machinery has been $1\frac{7}{16}$ in. However, at this length the maximum yarn strength which viscose staple is capable of yielding is apparently not obtained because the yarn strength rises in relation to increasing fiber length, and at $2\frac{1}{8}$ in. it is still rising. Additional strength-staple-length data suggest that for maximum-strength yarn optimum staple length is approximtely $2\frac{1}{2}$ in. (for "Fibro" of 1.50 den.). Denier-yarn strength figures indicate that yarn strength rises again in an approximately linear relation with fiber fineness, the strongest yarns yielding the finest fibers. In order to obtain a yarn of the highest strength, a combination of staple length of not less than $2\frac{1}{2}$ in. and the finest filament denier possible would be necessary. To command the full potentials of rayon staple, length-denier count-range relationships were investigated. While longer staples and finer deniers yield stronger yarns, it is also indicated that they increase the count range over which the material can be spun. A reasonable spinning limit established commercially for viscose rayon staple $1\frac{7}{16}$ in. long with a filament denier of 3 on cotton spinning machinery is single 20s cotton. In this case, an increase in length to $2\frac{1}{2}$ in. results in 30s cotton counts, and to 4 in., 40s cotton counts. Similar effects are found in other combinations of lengths and deniers. Hence there are 5 factors which can be set off one against the other to attain specific characteristics, namely, staple length, filament denier, yarn strength, yarn character, and count spinning limit. A review of existing spinning systems reveals one major difficulty—sliver-making in fine deniers. A radical approach to the solution of these limitations imposed on rayon staple is seen in the various methods in tow-to-top conversion. Unfortunately, the simultaneous breaking or cutting of the fibers results in an unsatisfactory fiber-length distribution. These difficulties have led to the consideration of a more difficult method of reducing tow to sliver by cutting, as distinct from breaking, thus insuring more nearly regular staple-length distribution. The result of this work is "Greenfield Top," a sliver of reasonable quality produced at a cost much below that of the conventional methods of sliver-making for worsted spinning. It has not, however, so far proved possible to provide satisfactory slivers in short lengths of, say, $2\frac{1}{2}$ or $3\frac{1}{2}$ in. in any denier, or lengths of 6 in. in the finer deniers such as 1.50, 1.25, and 1.00. From the standpoint of quality and cost in the shorter lengths up to 3 in. and finer deniers of 1.50 to 1.00, it is doubtful whether satisfactory slivers can ever be produced by this method. An alternative was sought in modification of existing opening, carding, and spinning mechanisms. Progress was noted in the revolving flat card and the roller and clearer cards, each with its own merits—the former performing satisfactorily on short lengths and fine deniers and the latter on coarser deniers. To overcome the limitations of drafting roller stands, modifications have usually taken the form of dispersing with 1 pair of the usual pairs of drafting rollers in the drawing frame, rearrangement of middle rollers in the 3-roller system of speed frames, and the application of slip-drafting methods in spinning frames. These cotton-spinning drafting arrangements have made possible the production of yarn from staples up to 3 in. in length and 3 den. and coarser. L. A. Fiori

Text. Research J. Mar. 1948

Spinning Limitations

Limiting factors for ring spinning frames. F. A. Westbrook. *Textile Manufacturer* **73**, 507–9 (Nov. 1947).

Abstract of a report by the Institute of Textile Technology concerning the factors of design inherent in spinning frames which limit both quality and production. The limitations imposed by the design or influence of the drafting rolls, roll stands, roving traverse motion, traveler speed and weight, ring size, spindle speed, size of package, and type of wind are discussed. The resonance set up by the change in nip of the front roll, due to the fluting of the bottom front roll, is discussed. It is claimed that the change in the position of the nip is one of the causes of irregular twisting. A. L. Landau

Text. Research J. Mar. 1948

Shuttle Control

Super shuttle control in weaving. Anon. *Text. J. Australia* **22**, 513, 532–3 (Sept. 20, 1947); *Text. Weekly* **38**, 114–8 (1946); and *Text. Mercury and Argus* **115**, 249–51 (1946).

An account of the "Macro" patent on shuttle swell, from the viewpoint of actual mill conditions, is given. It can be fitted to all types of looms and has already been widely adapted for weaving on cotton, rayon (viscose, Celanese, Bemberg), spun rayon, and linen looms. The author describes the advantages of this device, placing emphasis on the economies realized from reduced running costs and waste losses, and the possibility of using soft-spun filling and the largest mule twist-size cops (up to $8\frac{3}{4}$-in. length) for increasing cloth production.

Text. Research J. Mar. 1948 L. A. Fiori

Shuttle Swell

"Simplex" self-aligning swell. A new arrangement for loose reed motions. Anon. *Text. Mercury and Argus* **117**, 637, 639 (Nov. 7, 1947); *Textile Weekly* **40**, 848, 850 (Nov. 7, 1947); *Textile Re-*

corder **65**, 60, 65 (Nov. 1947); *Textile Manufacturer* **73**, 503 (Nov. 1947).

This development for shuttle checking consists of a leather-covered hardwood piece pivoted about 2 points so that it aligns itself to any angle. The special position of the fulcrum points and an adjustable spring pressing centrally on the back of the wooden swell itself bring the shuttle to rest with a minimum of shock. Advantages claimed are: (1) elimination of trapped filling on entry or exit of the shuttle, (2) directing of the shuttle toward the reed, irrespective of the picker position, (3) doubling of picker-stick life, (4) lessening of "sloughing-off" of filling, and (5) saving of power. At present it is being produced only for loose reed looms, but a fast reed type is now being tested. L. A. Fiori

Text. Research J. Mar. 1948

Loom Production

Increased production per man hour in weaving with particular reference to automatic looms. T. Joy. *J. Text. Inst.* **38**, 434–41 (Sept. 1947).

A discussion of the factors responsible for increased production per man hour on automatic looms. Consideration is given to environment, hereditary textile ability, number of looms per weaver, preparation of warps, etc. L. A. Fiori

Text. Research J. Mar. 1948

Building Motion for Speed Frames

A new building motion for speed frames. Anon. *Text. Mercury and Argus* **117**, 692 (Nov. 14, 1947): *Textile Weekly* **40**, 896 (Nov. 14, 1947).

This is a greatly improved type of building motion designed by Tweedales and Smalley Ltd. (1920), for application to their speed frames. Adjustments are facilitated and machine efficiency is improved by replacing the unreliable ratchet gear catch with a large range of spur-change gears. This makes possible very fine changes in bobbin circumferential speed, thus eliminating the risk of too slack or too tight winding which exists with the ratchet arrangement which does not lend itself to precise adjustments.

Text. Research J. Mar. 1948 L. A. Fiori

Weaving Mechanisms

"More Looms" system adapting the Lancashire loom. J. M. Yerkess. *Text. Weekly* **40**, 806, 808, 810 (Oct. 31, 1947); *Text. Mercury and Argus* **117**, 606–8 (Oct. 31, 1947).

This paper was read at the Buxton Conference, Oct. 11. It has particular reference to mechanisms which relieve the weaver of responsibility of supervision. Descriptions of multiple boxes for single-shuttle work, filling feeler motion and measuring motion, and other critical loom parts are given. Weaver task and load distribution are also discussed.

Text. Research J. Mar. 1948 L. A. Fiori

Warp Tension in Weaving

Wool yarn weaving, with particular reference to the controlling of warp tension. E. J. Poole. *J. Text. Inst.* **38**, P286–99 (Aug. 1947).

This discussion deals with a new development in warp tension control. The author reviews briefly weaving mechanisms and let-off motions with particular regard to the control of the warp from the beam. Because wools are susceptible to atmospheric changes and stretch more readily at high than at low humidities, the resistance of a wool warp to stretch will vary with moisture content. Therefore, against the constant load systems now generally in use, which are dependent upon the pull of the warp against a sensitive oscillating back rail, different degrees of stretch imposed on the warp eventually are passed on to the cloth, causing phases of relaxation and resulting in warpwise streakiness, gray-length variations, and many other cloth imperfections. A new method is proposed whereby the warp tension is controlled by means of 2 positively driven metal fluted rollers covered with an elastic oil-resistant rubber sleeve at about 10% tension and synchronized with the take-up mechanism. These rollers are intergeared so as to provide a cushion grip on the warp yarn. The degree of warp tension is measured by means of a let-off change gear which, when once determined for a particular cloth, remains constant. From the results of a series of tests carried out on a loom equipped with this improved type of roller let-off motion and running under ordinary mill conditions, it was concluded that warp being delivered at a constant rate will result in uniform piece lengths and the elimination of other cloth faults. Charted movements of the back rail relative to the angular movement of the beam of a common type of positive worm let-off motion are shown. Here can be seen plainly the varying degrees of tension set up in the warp during weaving and the warp stretch under the influence of the weighing system during standing periods. 14 patents on this new development are described briefly. L. A. Fiori

Text. Research J. Mar. 1948

Spooler Work Loads

Charts aid computing spooler's task. R. Leppla. *Textile World* **97**, 110–11, 244, 248 (Dec. 1947).

Method and formulas for calculating the work loads that should be established for spooling. Tables giving job loads and production figures for different types of yarn are reproduced. A. L. Landau

Text. Research J. Mar. 1948

Textile Processing

Postwar textile processing. R. W. Jacoby. *Am. Dyestuff Reptr.* **36**, P347–51, P364 (June 30, 1947).

A review of developments in equipment and processes which resulted largely from the tremendous volume of production required to fill military needs. Emphasis is on high-capacity, flexible, continuous-type ma-

chinery. Impact of war production on postwar demand for quality goods, and the changing distribution of operational costs which necessitates optimum efficiency, are also discussed. K. S. Campbell

Text. Research J. Mar. 1948

Yarn Manufacturing Systems

Continental and Anglo-Continental drawing. J. B. Wright. *Text. J. Australia* **22**, 530–2 (Sept. 20, 1947).

After a brief review of the Bradford (English) and Continental systems of yarn manufacture, the author discusses a combination of these systems called Anglo-Continental drawing. This modified porcupine system, first introduced by Stell in 1929, consists of pin control by a porcupine roll in the first 6 or 7 operations, followed by the English system of draft against twist as the thickness of the sliver becomes sufficiently reduced. Features of this system are the patent stripping device on the porcupine roller which prevents sticking and laps; the use of double rollers at the delivery end, enabling the porcupine to be set closer with better control of short fibers; the substitution of revolving funnels for the rubbing leathers which are unsuitable for oil-combed slivers; and the selection of the cone system of winding to permit working with the smallest amount of twist in the last 1 or 2 operations. The system is adaptable to a wide range of wools (2 in. to 9 in. long and from 70s to 50s quality) either oil- or dry-combed. Advantages over the English system are a reduction in waste, increased production, and manufacture of yarns having properties of both English and Continental systems. L. A. Fiori

Text. Research J. Mar. 1948

Yarn-Numbering Systems

Yarn numbering. D. C. Snowden. *Silk J. and Rayon World* **23**, 40–1, 59 (Feb. 1947).

The many direct and indirect yarn-numbering systems are given, in two tables, with respect to weight unit, length unit, and yds. per lb. of No. 1s yarn. The general advantages and disadvantages are discussed. A system based on "drams per thousand yards" is proposed as "an ideal universal counts system," and one more likely to be adopted by the textiles industries than the Grex, Poumar, or Typp systems.

Text. Research J. Mar. 1948 R. K. Worner

Clearer Types

Selection and maintenance of roll clearers on preparatory machinery. R. S. Crowsly. *Textile Age* **11**, 44, 48, 50, 52, 54 (Dec. 1947).

A discussion of the types of clearers used on preparatory machinery in the spinning mill. The number of rolls, the position of the rolls, roll-covering material and the character of the fibers being processed are factors affecting the performance of the clearer. Clearers on the card and the drawing frame are discussed. A. L. Landau

Text. Research J. Mar. 1948

MISCELLANEOUS

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Hot-Melt Coating

Butyrate hot-melt coatings. Martin Solo. *Modern Packaging* **20**, 127–8, 166, 168 (1947) (through *Chem. Abstr.* **41**, 7746*a* (Nov. 20, 1947)).

The newly developed hot-melt compositions, containing major amounts of low, η celulose acetate butyrate and minor proportions of plasticizers, resins, and waxes can be applied at high speeds without solvent hazards and yield protective packaging papers with high block temperatures. Properties and applications are discussed.

Text. Research J. Mar. 1948

Gray Goods Defects

Bothersome gray goods defects. Anon. *Am. Wool and Cotton Reptr.* **61**, 53, 55 (Dec. 18, 1947).

A digest of a speech concerning the irregularities in gray goods and their effect upon the quality of the finishing process. Among the defects which cause trouble are thin places in the cloth, slubs, hanging threads, and the presence of tramp metal on the cloth. A. L. Landau

Text. Research J. Mar. 1948

Chemical Resistance of Laminates

Chemical resistance of phenolic laminates. W. R. Tyrie. *Trans. Am. Soc. Mech. Engrs.* **69**, 795–7 (1947) (through *Chem. Abstr.* **41**, 7812*a* (Nov. 20, 1947)).

Eight representative types of phenolic laminates were immersed in various aqueous solutions (1 and 20% HCl, 1 and 10% NaOH, 1% chromic acid, and a solution such as is encountered in the rayon-spinning industry) for 6 mos. and tested periodically for weight change, dimensional change, and strength losses. On the whole any laminate which has the desired properties after prolonged immersion in water alone (this eliminates paper-base laminates) would also be satisfactory for other field conditions, except strong acid or any alkaline solutions. Where alkaline or strong acid conditions are involved, special care must be taken in the choice of the laminate. Glass-base laminates are especially unsatisfactory under strongly alkaline conditions; cotton-base laminates give good service under these conditions. For strongly acid conditions, a laminated material should be used only in special cases in which investigation has shown important advantages.

Text. Research J. Mar. 1948

Machine Lubrication

The lubrication of textile machinery. Juan B. Puig. *Acero y energia* **4**, 42–9 (138–45) (1947) (through *Chem. Abstr.* **41**, 7715*c* (Nov. 20, 1947)).

The constitution, properties, and application of lubricating oils used in the textile industry are discussed.

Text. Research J. Mar. 1948

Paper Binder Twine

Sulfite spinning paper for binder twine. Rudolf Trenschel. *Das Papier* **1**, 8–16 (1947) (through *Chem. Abstr.* **41**, 7748*g* (Nov. 20, 1947)).

Trenschel reviews the manufacture of spinning papers in Germany and the change from sulfate to a mixture of sulfate-sulfite and then to sulfite pulps. The folding endurance was found to be a suitable criterion of the value of a sulfite paper for this purpose. Uniformity of the strength properties is very important. The final minimum specifications for sulfite paper were: breaking length 9,000 and 3,000 mm. in machine and cross-machine directions; elongation, 1.6 and 2.5%; folding endurance, 2,000 and 200 folds; water absorptivity, 45–60%; wet strength, 15% of dry strength. The spinning of paper into binder twine is discussed.

Text. Research J. Mar. 1948

Theory of Polymer Structure

Statistical theory of rubberlike substances. Ryogo Kubo. *J. Colloid Sci.* **2**, 527–35 (Nov. 1947).

A theory of elasticity and double refraction of network structures formed by linear polymers is presented. It differs from theories published (outside of Japan) on some fundamental points. No new data. E. D. Klug

Text. Research J. Mar. 1948

Laundering Rayon

Mechanical degradation of rayon fabrics in domestic laundry procedures. M. H. Graydon, D. M. Lindsley, and J. B. Brodie. *Am. Dyestuff Reptr.* **36**, 397–9 (July 14, 1947).

Five types of rayon fabrics were given 2 series of 50 machine-washing treatments and 2 series of 50 hand-washing treatments using Zeolite-softened water in a 0.2% neutral soap solution at 100°–105°F. No one method produced significantly greater tensile strength losses than any of the others; repeated washing did cause a slight decrease in strength. Results indicated that present-day rayon fabrics may withstand more vigorous laundering methods than are now recommended. The 3 harshest of the 4 home methods tested—soaking, machine washing, and machine wringing—produced no greater degradation than any of the other 3 methods and proved to be the most efficient in soil removal. For white rayon fabrics similar to those studied, a preliminary soaking is recommended. Fabrics tested were acetate satin, acetate taffeta, viscose taffeta, viscose spun slub crepe, and cuprammonium crepe. Authors

Text. Research J. Mar. 1948

Identification of Resins

Individual characteristics of plastics. W. J. Nijveld. *Verfkroniek* **20**, 191–4 (1947) (through *Chem. Abstr.* **41**, 7811*f* (Nov. 20, 1947)).

Qualitative reactions comprising the Storch-Morawski reaction, saponification with absolute alcohol-KOH, Lassaigne or Kappelmeier test for N-containing resins, phenolic resin reaction of Kappelmeier, odor upon burning, *n*, and spot test with concentrated H_2SO_4 are carried out on 47 resins used for plastics. The results are summarized in a large, complete table. 8 references.

Text. Research J. Mar. 1948

Selvage Imperfections

Selvage imperfections. G. Creasy. *Textile Age* **11**, 59–60, 62, 64 (Dec. 1947).

The first article of a series on the maladjustments of the loom which will cause the formation of poor selvages. The type of fur used on the shuttle must be suitable for the yarn being woven. The checking and picking of the shuttle must be properly adjusted to eliminate poor selvages. A. L. Landau

Text. Research J. Mar. 1948

Burlap Economics

Burlap. Report no. 26, war changes in industry series. U. S. Tariff Commission, Washington, U. S. Govt. Printing Office, 1947. 58 pages. Price, 20 cents.

Imports of burlap into the United States exceed imports of cotton or wool manufactures in value and weight, averaging 458,000,000 lbs. annually since 1930. A survey in 1939–40 showed that ¾ of our burlap imports were made into bags, and the remainder used mostly as linings and wrappings, balings for textiles, cordage, etc. India's export tax, which is much higher on raw jute than on burlap, and her low wages, averaging less than $2.10 per wk., have discouraged burlap manufacture in the United States. India is said to be able to meet fully the world's long-time burlap requirements. Tariff and other government controls, world marketing, and transportation are discussed.

Text. Research J. Mar. 1948 R. B. Evans

Costing in Cotton Mills

Cost knowledge is vital in growth toward efficiency. Lewis F. Sawyer. *Textile World* **97**, 155, 202–6, 209 (Nov. 1947).

Only about half of the cotton mills are said to have used adequate cost accounting methods in 1946, yet these methods are essential in determining selling prices and for profitable operation. The Cotton-Textile Institute has prepared a series of cost manuals. R. B. Evans

Text. Research J. Mar. 1948

Brand Names in Textile Industry

Brand names for profit. *Am. Wool and Cotton Reptr.* **61**, 1–106 (Oct. 30, 1947).

Various aspects of the use of brand names are given in 26 articles in this special issue. R. B. Evans

Text. Research J. Mar. 1948

Textile Film Index

Textile films. Anon. *J. Soc. Dyers and Colourists* **63**, 345–50 (Nov. 1947).

A list of picture films of interest to the textile field which are available

in the United Kingdom is given. The film distributors, the size, and running time are listed, along with a short description of each film. Over 100 films. J. A. Woodruff

Text. Research J. Mar. 1948

Research Problems Involving Textiles

Research areas of textiles and clothing. Muriel Brasie, Margaret L. Brew, Cleo Fitzsimmons, Marjorie Rankin, and Russell C. Smart. *J. Home Econ.* **39**, 620–4 (Dec. 1947).

Research problems involving textiles and clothing as related to economics, psychology, and sociology are summarized. R. B. Evans

Text. Research J. Mar. 1948

PATENT REFERENCES

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Cotton Picker

Cotton harvesting. August E. Zock. U. S. 2,430,202 (Nov. 4, 1947).

A device for picking cotton by means of power-actuated equipment embodying pneumatic principles. A semicircular head or nozzle, having an overhanging flare screen to facilitate thrusting through the foliage, is operated manually by an operator who places it in the vicinity of the cotton bolls. A suction fan draws the fibers into a separator chamber, where the extraneous matter is pulled through a series of narrow slots formed by a network of narrow slats spaced about $\frac{1}{16}$ in. apart. As the good fibers accumulate on the bottom surface of these slats, they are wiped off by rotating flexible blades into a duct and then exhausted into a receiving bag. In the meantime, the waste is conveniently discharged on the ground beside the rows of cotton so that the still-growing crop is not contaminated nor are the operators disturbed. L. A. Fiori

Text. Research J. Mar. 1948

Dye Assistant

Dyeing fabrics containing cellulosic material and cellulosic material treated with quaternary nitrogen compounds. Glenn F. Womble and Paul Feldmann (to Dan River Mills). U. S. 2,430,153 (Nov. 4, 1947).

Cellulosic materials normally having an affinity for direct cotton dyes are treated with chemicals identified by the following general formula:

$$R—X—CH_2—N(\text{tert})—Y$$

in which X stands for oxygen, or for CO—N—R′ where R' represents hydrogen or an aliphatic hydrocarbon radical; R stands for an aliphatic hydrocarbon radical of at least 5 carbon atoms when X stands for CO—N—R′, and for an aliphatic hydrocarbon radical of at least 12 carbon atoms when X stands for oxygen; said carbon chains may be normal or branched, may or may not contain double bonds, or may be of a more complicated structure; N (tert) stands for a tertiary amine that may be aliphatic or heterocyclic, and Y stands for halogen or for an anion of a water-soluble acid. After the application of such chemicals, the treated material is dried and heated. As a result the cellulosic material loses its affinity in varying degrees for the direct cotton dye and acquires an affinity for dispersed dyes such as are normally used for dyeing cellulose acetate. J. A. Woodruff

Text. Research J. Mar. 1948

Crinkled Fabrics

Crinkled fabric and method for producing same. Albert Sydney Jones and Gerald B. Stackpole (to Cranston Print Works Co.). U. S. 2,429,935 (Oct. 28, 1947).

Fabrics capable of wet stretching such as that obtained in continuous bleaching operations and also capable of relaxation shrinkage are made to form wrinkle effects by printing the stretched fabrics with a permanent resist and then forming the pucker by shrinkage of the unprinted portions through normal relaxation. The original length of the cloth prior to stretching is then retained. J. A. Woodruff

Text. Research J. Mar. 1948

Multiple Fabrics

Weaving and woven fabrics. Edward Glendinning and Frank Binns. U. S. 2,424,928 (July 29, 1947).

Essentially, the fabrics manufactured by this method belong to the double and triple cloth family of weaves except that folds, ribs, or piles of pronounced amplitude are woven on one or both sides of the ground fabric. This results in a relatively thick cloth, suitable for padding, fireproofing (asbestos or material rendered fireproof by treatment can be woven into it), and novel effects such as a continuous pilelike cloth surface. Features of the apparatus are a ground-forming warp controlled by the usual type of let-off motion, a fold-forming warp provided with a special friction let-off system, a mechanism for interrupting the take-up of the cloth, and a control connection regulated from the dobby head for easing tension of the back rollers over which the folding warp passes.

Text. Research J. Mar. 1948 L. A. Fiori

Rib Fabric

Woven fabric and method of making same. Peter A. Preneta (one-third to Louis de Marco), U. S. 2,424,771 (July 29, 1947).

This invention is intended to provide a fabric in which a series of closely grouped and upstanding ribs are woven on the upper face of a ground or body weave without requiring the use of wires. The loops of the ribs are firmly tied together and to the body, producing a strong and long-wearing fabric. The basic structure of this fabric consists of 4 picks on top and 2 on the bottom—i.e., 2 picks on the ground or body weave alternate with 4 picks on the face of the ground or body, forming the closely adjacent ribs. L. A. Fiori

Text. Research J. Mar. 1948

Setting Twist

Method of treating twisted filamentary materials. Hayden B. Kline and Alden H. Burkholder (to Industrial Rayon Corporation). U. S. 2,421,334 (Apr. 26, 1947); U. S. 2,421,336 (May 27, 1947).

The twist of air-dry twisted nonmetallic filamentary materials is set by heating wound packages dielectrically in a high-frequency electrical field. Even when relatively large yarn packages are used, the twist will be set in a uniform manner. In general, this apparatus includes a pair of spaced electrodes connected to a source of high-frequency power and means for passing the material into the electrical field between the electrodes. L. A. Fiori

Text. Research J. Mar. 1948

Stop Motion

Stop motion for knitting machines and the like. Allan Clair Thomas (to Celanese Corporation of America). U. S. 2,421,092 (May 27, 1947).

This invention provides a stop motion that can be used for knitting machines, looms, inspection tables, and particularly for warp knitting machines. An increase or decrease of light intensity transmitted to a photoelectric cell by a missing yarn or a double yarn in the fabric causes the motion to become operative either by stopping the machine or working a visible or audible signal. This is an improvement over the inventor's previous patent, U. S. 2,346,240, whose undesirable contact milliammeter switch has been replaced by a reliable light valve current meter. L. A. Fiori

Text. Research J. Mar. 1948

Synthetic Protein Fibers

Shaped protein structures and their preparation. Gordon F. Biehn and Edward T. Cline (to E. I. du Pont de Nemours & Company). U. S. 2,429,214 (Oct. 21, 1947).

Prolamines are converted into synthetic fibers and similar filamentary articles by extruding an aqueous alkaline solution of, e.g., 12 to 18% zein into an aqueous coagulating bath containing formaldehyde, strong mineral acid, and inorganic salts. The filaments so formed are partially hardened with an aqueous solution of a protein tanning agent and soluble inorganic salts, and then stretched at least 50% of their maximum draw ratio while passing through a solution of water-soluble neutral and acid-reacting salts of mineral acids at temperatures in excess of 50°C. The stretched filaments are hardened in an aqueous formaldehyde solution. Zein filaments thus prepared have improved wet and dry strength as well as high elastic recovery, and show an orientation number of 1.67 as measured by x-rays. J. A. Woodruff

Text. Research J. Mar. 1948

Heat-Sealing Without Discoloration

Heat-sealing moistureproofing coatings. James A. Mitchell (to E. I. du Pont de Nemours & Co.). U. S. 2,430,726 (Nov. 11, 1947).

OPPORTUNITY FOR RESEARCH CHEMIST

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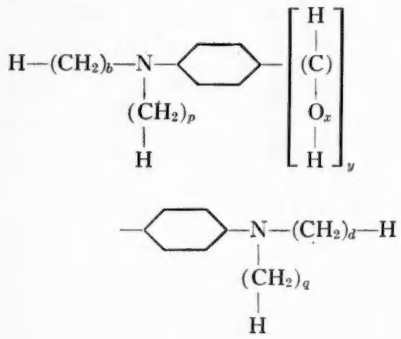
The discoloration occurring when iratol, a product obtained by treating beta-napthol with rubber, is used as a heat-sealing cementing agent for cellulose sheet is obviated and the life of the cementing material is lengthened by the incorporation of substances comprehended by the general structural formula:



in which b, d, p, and q are numerals from the group consisting of 0, 1 and 2, and x and y are numerals from the group consisting of 0 and 1. J. A. Woodruff

Text. Research J. Mar. 1948

Shrinkproofing Wool

Antishrinking treatment of animal fibers. Francis Malcolm Stevenson and John Leonard Raynes (to Stevensons Ltd.). U. S. 2,429,082 (Oct. 14, 1947).

The tendency of wool to shrink when washed is reduced by treatment of the wool with an aqueous solution of a permanganate compound and with either an aqueous solution or suspension of a nitrogen-chloro compound or with an aqueous solution or suspension of an alkali metal or alkaline earth metal hypohalite. The nitrogen-chloro compounds are defined as those in which the chlorine atom is directly attached to the nitrogen atom and wherein the chlorine is available chlorine. The use of the permanganate as an oxidizing agent has the effect of making the animal fibers more susceptible to attack by the halogen-containing agent. The action of the halogen takes place at a pH from 7.5 to 11, which allows slow reaction of the halogen and the wool. The final treatment is with an acidified bisulfite to remove discoloration due to the permanganate. The weight loss of wool during the treatment is low and the natural properties of handle and resilience are retained. J. A. Woodruff

Text. Research J. Mar. 1948

Wool Grease

Treatment of wool-washing effluent. Frank Totney (to Industrial Development Corp. of South Africa, Ltd.). U. S. 2,421,094 (May 27, 1947).

This method presents a process by which wool grease is separated from wool scouring liquor, and also a means of disposing of the wool washing effluent without objectionable pollution of sewage channels, streams, etc. Evaporation of the effluent is continued until a content of about 4 to 5% of wool grease is obtained; the concentrated effluent is then heated to a temperature of from about 290 to 302°F and a gage pressure of about 50 lbs. per sq. in., and the treated effluent is settled for about 1 to 8 hrs. to separate the grease, suint, and muddy liquor from each other. L. A. Fiori

Text. Research J. Mar. 1948

Carpet Pile Yarn

Process of making pile fabric floor covering. Robert J. Jackson (to Bigelow-Sanford Carpet Co., Inc.). U. S. 2,430,425 (Nov. 4, 1947).

A carpet in which the pile has the curly appearance of Persian lamb is produced. Worsted roving (25 grains per yd. and 1.8 turns per in.) is twisted on a spinning frame, forming hard, twisted yarn (between 5 to 10 turns per in.). After being reeled into skeins and collapsed into a tensionless kinky condition, this yarn is set by wetting with water and treating with saturated steam. It is then dried and wound on bobbins. Finally, twist (from 1 to 4 turns per in.) opposite to the original direction is inserted. The yarn, now soft-twisted, is ready to be woven as the pile yarn of a carpet fabric. Owing to the nature of the twisted yarn, the pile loops are limited in number (8 to 20 per sq. in.) compared to the number (as many as 75) in an ordinary pile carpet fabric. Worsted yarns are preferred but woolen yarns may also be used. L. A. Fiori

Text. Research J. Mar. 1948

Yarn Breakage Detector

Yarn breakage detector. William D. Kellog and Richard F. Eshleman (to Aveco, Inc.). U. S. 2,427,055 (Sept. 9, 1947).

An arrangement is described for detecting yarn breakage on winding and twisting machines, which is especially valuable on tricot or warp knitting machines, beaming machines, and looms. Basically, the unit consists of 2 separable electroconductive members (with at least one mounted pivotally) arranged side by side and normally in contact except when yarn passes between them. Breakage of the yarn allows the members to make contact which results in a stoppage of the machine through an electric relay. Since the invention does not rely on gravity or on relatively shiftable parts, a claim is made that it affords great reliability, durability, and less servicing. L. A. Fiori

Text. Research J. Mar. 1948

Impregnation of Fibrous Webs

Apparatus for producing saturated fibrous bodies. Joshua H. Goldman (to Fiber Products Laboratories, Inc.). U. S. 2,429,314 (Oct. 21, 1947).

Improved saturating conditions are obtained on fibrous webs. The high-viscosity saturating liquid is picked up by one or more embossed rollers and the web is caused to pass between two such rollers under pressure, with a resultant even saturation. Another feature is the reduced tendency of the web to break and wrap around the embossed rollers as compared with the same action on nonembossed rollers. J. A. Woodruff

Text. Research J. Mar. 1948